FORM I INORGANIC ANALYSIS DATA SHEET

Lab Name: Northern Lake Service, Inc

SDG No.:(project) ~~56054~~, 56058,

Lab Sample I.D.: See final report

Matrix (soil/water): (S) W

Date Received: See final report

% Solids See final report if applicable

Date analyzed: 10-2-2000

FORM 2A INITIAL AND CONTINUING CALIBRATION VERIFICATION

Initial Calibration Source: High Purity #023532 Continuing Calibration Source: Spex #23-117MM

Concentration Units: mg/L

Analyte	Initial Calibration			Continuing Calibration					
	True	Found	%R(1)	True	Found	%R(1)	Found	%R(1)	M
Aluminum	500 mg/L	505.1	101	10 mg/L	1 10.36	104%	2 10.69	107%	EPA 6010
					3		4		EPA 6010
					5		6		EPA 6010
					7		8		EPA 6010
					9		10		EPA 6010

FORM 2B LOD STANDARD FOR AA AND ICP not applicable

FORM 3 BLANKS

Preparation Blank Matrix (soil/water): (S) W Preparation Blank Concentration Units (mg/L)

Analyte	Init.Calib. Blank		Continuing Calibration Blank (mg/L)						Preparation Blank		
	(mg/L)	Q	CCB #	Q	CCB #	Q	CCB #	Q	Batch #	R	Q
Aluminum	ND		1 ND		2 ND		3		39	.0692	
			4		5		6		47	ND	
			7		8		9				
			10		11		12				

FORM 4 ICP INTERFERENCE CHECK SAMPLE not applicable

FORM 5A SPIKE DUPLICATE

Matrix (soil/water): (S) W Level : see below Concentration Units (mg/L)

Analyte	Sample Number	Spike Sample Result (SSR)	Q	Sample Result (SR)	Q	Spike Added (SA)	%R	Q	Method
Al instr	238341			91.73		50.0	95%		EPA 6010
Al dig									EPA 6010
Al instr	238404			128		50.0	90%		EPA 6010
Al dig	238404			128		10.0	0%		EPA 6010
Al instr									EPA 6010
Al dig									EPA 6010
Al instr									EPA 6010
Al dig									EPA 6010
Al instr									EPA 6010
Al dig									EPA 6010

FORM 7 LABORATORY CONTROL SAMPLE (LFB)

Aqueous LCS Source: High Purity NLS 1 Lot #015206

Analyte	Aqueous (mg/L)				Aqueous (mg/L)			
	True	Found	%R	Batch #	TRUE	Found	%R	Batch #
Aluminum	10.0	10.43	104%	39	10.0			
Aluminum	10.0	9.560	96%	47	10.0			
Aluminum	10.0				10.0			
Aluminum	10.0				10.0			

500120

FORM 9 ICP SERIAL DILUTIONS not applicable

FORM 10 Method Detection Limits

ICP ID Number: TJA A-25 Date: see below

Analyte	Wave Length (nm)	Back-ground	LOD (mg/L)	LOQ (mg/L)	Method	Date
Al	308.215	automatic	0.0341	0.121	EPA 6010	1/26/00
Ag	328.068	automatic	0.0094	0.037	EPA 6010	3/3/00
As	189.042	automatic	0.29	1.03	EPA 6010	4/27/00
Ba	455.403	automatic	0.005	0.005	EPA 6010	2/23/00
Ca	315.877	automatic	0.3 (R.L.)	0.3 (R.L.)	EPA 6010	NA
Cd	214.438	automatic	0.0064	0.025	EPA 6010	2/23/00
Cr	267.716	automatic	0.018	0.071	EPA 6010	2/23/00
Cu	324.754	automatic	0.0061	0.024	EPA 6010	2/23/00
Fe	259.94	automatic	0.046	0.181	EPA 6010	2/23/00
K	766.49	automatic	0.406	1.6	EPA 6010	2/23/00
Pb	220.353	manual	0.164	0.644	EPA 6010	2/23/00
Mn	257.61	automatic	0.0097	0.038	EPA 6010	2/28/00
Na	589.592	automatic	0.0439	0.173	EPA 6010	2/23/00
Ni	231.604	automatic	0.02	0.079	EPA 6010	2/28/00
Zn	213.856	automatic	0.012 (R.L.)	0.012 (R.L.)	EPA 6010	NA

FORM 11A ICP INTERELEMENT CORRECTION FACTORS (ANNUALLY)

ICP ID Number: TJA A-25 / Date: see below

Analyte	Wavelength (nm)	Interelement Correction Factors for: Al	Ca	Fe	Mg	Cr	Date
Al	308.215	NA	NA	NA	NA	NA	1/26/00
Ag	328.068	NA	NA	NA	NA	NA	2/23/00
As	189.042	NA	NA	NA	NA	0.0004323	4/27/00
Ba	455.403	NA	NA	NA	NA	NA	2/23/00
Ca	315.877	NA	NA	NA	NA	NA	NA
Cd	214.438	NA	NA	NA	NA	NA	2/23/00
Cr	267.716	NA	NA	NA	NA	NA	2/23/00
Cu	324.754	NA	NA	NA	NA	NA	2/23/00
Fe	259.94	NA	NA	NA	NA	NA	NA
K	766.49	NA	NA	NA	NA	NA	2/23/00
Pb	220.353	0.000612	NA	NA	NA	NA	2/23/00
Mn	257.61	NA	NA	NA	NA	NA	2/28/00
Na	589.592	NA	NA	NA	NA	NA	2/23/00
Ni	231.604	NA	NA	NA	NA	NA	2/28/00
Zn	213.856	NA	NA	NA	NA	NA	NA

FORM 11B ICP INTERELEMENT CORRECTION FACTORS (ANNUALLY) see above

FORM 12 ICP LINEAR RANGES

ICP ID Number: TJA A-25 Date: see below

Analyte	Integ. Time (Sec.)	Concentration (mg/L)	Method	Date
Al	2	500	EPA 6010	1/26/00
Ag	2	1	EPA 6010	8/3/00
As	2	100	EPA 6010	4/27/00
Ba	2	20	EPA 6010	8/3/00
Ca	2	200	EPA 6010	2/23/00
Cd	2	20	EPA 6010	8/3/00
Cr	2	20	EPA 6010	8/3/00
Cu	2	20	EPA 6010	2/23/00
Fe	2	200	EPA 6010	2/23/00
K	2	200	EPA 6010	2/23/00
Pb	2	20	EPA 6010	8/3/00
Mn	2	20	EPA 6010	2/28/00
Na	2	200	EPA 6010	2/23/00
Ni	2	20	EPA 6010	2/28/00
Zn	2	20	EPA 6010	2/23/00

320 -- Aluminum, tot. as Al

PAGE: 1

PRINTED: 10/02/00 09:29:26

TYPE: Metal (I) ID: 21

DATE 10-2-00
ANALYST JAC
CHECKED TR
ENTERED
SPK CON
VER DATE
STD DATE

LOD: (NON) 0.019 mg/L 08-JUN-98
LOQ: (NON) 0.068 mg/L 08-JUN-98
LOD: (DIG) 0.034 mg/L 26-JAN-00
LOQ: (DIG) 0.12 mg/L 26-JAN-00

HOLDTIME: 21 days
PAL: ES:

METHODS:
____ SOP#
X EPA EPA 200.7
____ SW846 SW846 6010
____ ASTM SM 3120B
____ NLS

MH = 21 S 2

MX	TYPE	UNITS	LOVAL	HIVAL	UCL	MEAN	LCL	DATE
C	DUP	mg/l	0	1	20.00		.00	
C	DUP	mg/l	1	5	20.00		.00	
C	DUP	mg/l	5	100	20.00		.00	
C	SPK	%	0	200	113.26	99.61	85.97	15-MAR-95
LCS	DUP	mg/l	0	1	20.00		.00	
LCS	DUP	mg/l	1	5	20.00		.00	
LCS	DUP	mg/l	5	100	20.00		.00	
LCS	SPK	%	0	200	120.00		80.00	
S	DUP	mg/kg	0	1	20.00		.00	
S	DUP	mg/kg	1	5	20.00		.00	
S	DUP	mg/kg	5	100	20.00		.00	
S	SPK	%	0	200	120.00		80.00	
SC	DUP	mg/l	0	1	20.00		.00	
SC	DUP	mg/l	1	5	20.00		.00	
SC	DUP	mg/l	5	100	20.00		.00	
SC	SPK	%	0	200	120.00		80.00	
W	DUP	mg/l	0	1	20.00		.00	
W	DUP	mg/l	1	5	20.00		.00	
W	DUP	mg/l	5	100	20.00		.00	
W	SPK	%	0	200	119.07	99.34	79.60	12-SEP-97

BLANK (BLK) - CHECKSTANDARD (CHK) - DUPLICATE (DUP) - RECOVERY (SPK) DATA

SAMPLE#	MTRX	TYPE	VALUE1	VALUE2	UNITS	WHO	DATE
238341	W	DUP	93.58	93.09	mg/l	JAC	10-2-00
11			95				
238404		SPK	108.8	112-1			
11			90				

EPA

FQL

500122

320 -- Aluminum, tot. as Al PAGE: 2

PRINTED: 10/02/00 09:29:26 DATE & TIME ANALYZED 10-2-00

DEFAULT UNITS mg/L mg/L mg/kg DWT

95%

238341 -- 56051 1 [redacted] 39 91.73 (2) 93.58 (3) 93.09 QC x 376
MAX: 14-SEP-00 – Soil, SD208006.5 – MTX= SO – 39 d-old 38 d-hse 09:17

238342 -- 56051 4 [redacted] 39 86.76 QC x 331
MAX: 14-SEP-00 – Soil, SD208007.5 – MTX= SO – 39 d-old 38 d-hse 09:42

238398 -- 56058 5 [redacted] 47 79.63 QC x 7439
MAX: 15-SEP-00 – Soil, SD207000.5 – MTX= SO – 38 d-old 35 d-hse 08:36

238399 -- 56058 6 [redacted] 47 92.40 QC x 6,571
MAX: 15-SEP-00 – Soil, SD207002.5 – MTX= SO – 38 d-old 35 d-hse 08:55

238400 -- 56058 7 [redacted] 47 85.91 QC x 6,905
MAX: 15-SEP-00 – Soil, SD207002.5/D – MTX= SO – 38 d-old 35 d-hse 08:55

238401 -- 56058 8 [redacted] 47 67.04 QC x 5196
MAX: 15-SEP-00 – Soil, SD207004.5 – MTX= SO – 38 d-old 35 d-hse 09:40

238402 -- 56058 9 [redacted] 47 111.5 QC x 3740
MAX: 15-SEP-00 – Soil, SB207006.5 – MTX= SO – 38 d-old 35 d-hse 10:23

238403 -- 56058 10 [redacted] 47 174.4 QC x 5843
MAX: 15-SEP-00 – Soil, SB207008.5 – MTX= SO – 38 d-old 35 d-hse 10:41

238404 -- 56058 11 [redacted] 47SD 128 (12) 108.8 90% (13) 112:1 (14) 104.6 (15) 118.2 QC x 3,64
MAX: 15-SEP-00 – Soil, SB207010.5 MS/ – MTX= SO – 38 d-old 35 d-hse 10:57

238405 -- 56058 16 [redacted] 47 89.03 17 BLK 18 STD QC x 3491
MAX: 15-SEP-00 – Soil, SB207012.5 – MTX= SO – 38 d-old 35 d-hse 11:26

238407 -- 56058 19 [redacted] 47 66.90 QC x 1731
MAX: 15-SEP-00 – Soil, SB207014.5 – MTX= SO – 38 d-old 35 d-hse 11:45

238408 -- 56058 20 [redacted] 47 85.0 QC x 2585
MAX: 15-SEP-00 – Soil, SB207015 – MTX= SO – 38 d-old 35 d-hse 12:05

238409 -- 56058 21 [redacted] 47 96.03 QC x 2,550
MAX: 15-SEP-00 – Soil, SB207015/D – MTX= SO – 38 d-old 35 d-hse 12:05

238642 -- 56099 [redacted] 47
MAX: 18-SEP-00 – Soil, SD215000.5 – MTX= SO – 35 d-old 33 d-hse 11:31

238643 -- 56099 [redacted] 47D
MAX: 18-SEP-00 – Soil, SD215002.5 – MTX= SO – 35 d-old 33 d-hse 11:56

238644 -- 56099 [redacted] 47D
MAX: 18-SEP-00 – Soil, SD215004.5 – MTX= SO – 35 d-old 33 d-hse 12:13

238645 -- 56099 [redacted] 47D
MAX: 18-SEP-00 – Soil, SB215006.5 MS/ – MTX= SO – 35 d-old 33 d-hse 16:45

238646 -- 56099 [redacted] 47
MAX: 18-SEP-00 – Soil, SB215008.5 – MTX= SO – 35 d-old 33 d-hse 16:49

500123

238647 -- 56099 [redacted] 47SD
MAX: 18-SEP-00 – Soil, SB215010.5 – MTX= SO – 35 d-old 33 d-hse 17:02

23 – PB 39 24 PB – 47 26 – INF 28 – BLK
23 – MB 39 25 MB – 47 27 – ICS 29 – STD

Method Status

Type: Quantitative (Standard Analysis)

Date Created : 04/30/99 21:42
Date Modified: 10/02/00 12:44

Number of Elements: 1 Number of Lines: 1

Lines Calibrated : 0
Lines Standardized : 1 10/02/00 09:24

Analysis Preferences

Number Repeats: 4
Delay Time: 0.0 Sample Flush Time: 40.0

Auto-inc Samplenames: Yes
Use Sample Weight Corrections: No
Use Manual Plasma Conditions: No
Collect Quick Scans: Yes

Preprocess Command File : None
Postprocess Command File : None

Automated Output

SAMPLETYPE	AUTOSTORE	AUTOPRINT	AUTOVIRTUAL	AUTOEXPORT
Unknowns	Yes	Yes	Yes	Yes
QCs	Yes	Yes	Yes	Yes
Blanks	Yes	Yes	Yes	Yes
Standards	Yes	Yes	Yes	Yes
Recoveries	Yes	Yes	Yes	Yes

Database : ANADATA Include Repeats: Yes Include Scan: No
Virtual File: results.dat (Overwrite)
Export File : results.txt (Append)

Autoprint Standardization Report -
Slopes: No Readbacks: No Plots: No

Report Preferences

500124

	UNKNOWN	BLANK	QC STD	STANDARD	RECOVERY
Output mode	Conc	Conc	Conc	IRatio	Conc
Print limits	No	No	No	-	No
Significant figures	Yes	Yes	Yes	Yes	Yes
Blank subtract	No	No	No	-	No
Line switch	Yes	No	Yes	-	Yes
IECs	Yes	Yes	Yes	-	Yes
Correction Factor	1	1	1	-	1
Perform Check	No	No	Yes	-	Yes
Check Table		BLCTAB	QCTAB	-	ROCTAB

Report To SCREEN -					
Averages	Yes	Yes	Yes	Yes	Yes
Statistics	Yes	Yes	Yes	Yes	Yes
Repetitions	Yes	Yes	Yes	Yes	Yes
Errors	Yes	Yes	Yes	Yes	Yes
Checks	No	No	Yes	No	Yes
Wavelength	No	No	No	No	No
Internal Standard Ref.	No	No	No	No	No
Units	No	No	No	No	No
Report To PRINTER -					
Averages	Yes	Yes	Yes	Yes	Yes
Statistics	Yes	Yes	Yes	Yes	Yes
Repetitions	Yes	Yes	Yes	Yes	Yes
Errors	Yes	Yes	Yes	Yes	Yes
Checks	No	No	Yes	No	Yes
Wavelength	Yes	Yes	Yes	Yes	Yes
Internal Standard Ref.	No	No	No	No	No
Units	Yes	Yes	Yes	Yes	Yes
Report To EXPORT -					
Averages	Yes	Yes	Yes	Yes	Yes
Statistics	Yes	Yes	Yes	Yes	Yes
Repetitions	Yes	Yes	Yes	Yes	Yes
Errors	Yes	Yes	Yes	Yes	Yes
Checks	No	No	Yes	No	Yes
Wavelength	Yes	Yes	Yes	Yes	Yes
Internal Standard Ref.	No	No	No	No	No
Units	Yes	Yes	Yes	Yes	Yes

Plasma Conditions

Torch Flow: High Flow
Auxiliary Gas: Medium

Flush Pump Rate (rpm): 100
Relaxation Time (sec): 0.0

	Group 1	Group 2	Group 3	Group 4	Special
RF Power (W):	1150	1350	950	1550	1150
Nebulizer Pressure (PSI):	36.0	36.0	36.0	36.0	36.0
Observation Hgt (mm):	n/a	n/a	n/a	n/a	n/a
Analysis Pump Rate (rpm):	100	100	100	100	100

Internal Standards

ELEMENT	FACTOR	ANALYTE
None	1.0	n/a

Al 308.215

Internal Std: None

Report information

Element Name	: Al3082	IEC Priority	: 0
Print units (conc)	: ppm	Sign. Figures	: 4
Lineswitch Low	: 10.00	Lineswitch High	: 500.00
Print Limit Low	: 0.00	Print Limit High	: 0.00
Raw factor	: 0.00	Conc. factor	: 1.00

Standards (2) Multipoint Linear

Name	Concentration	Signal(IR)
Blank	0	.030596
AL500	500	136.718

IECs (0) Use IECs? No

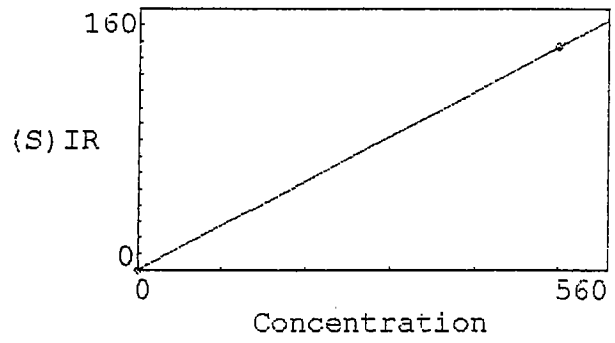
Sequential Acquisition
Integration Time: 2.0
Plasma Group: 1
WL Locate Method: Normal
Slit Height: 3
Prescan Window Size: Default
Peak Offset: 0.0051
High Voltage: 674

Background : Automatic

El Name	Slope	Y-int	Correlation	Date Stdized
Al3082	0.2734	0.0306	1.0000000	10/02/00 09:24:00

Standard	Concentration		Difference		Signal
Name	Stated	Found	Conc	%	(S)IR
Blank	0	0	0	0	0.030596
AL500	500	500	0	0	136.72

Al 308.215



Method: ALTEST Sample Name: AL500 Operator:
Comment:
Run Time: 10/02/00 09:16 Type: Std Mode: IR Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	Cts
Avg	136.7
Stddev	.8
%RSD	.5872
#1	135.8
#2	137.7
#3	136.7
#4	136.7

Method: ALTEST Sample Name: Blank Operator:
Comment:
Run Time: 10/02/00 09:22 Type: Std Mode: IR Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	Cts
Avg	.0306
Stddev	.0060
%RSD	19.47
#1	.0270
#2	.0267
#3	.0394
#4	.0293

Analysis Report 10/02/00 09:26:46 page 1

Method: ALTEST Sample Name: al 500 Operator:
Comment:
Run Time: 10/02/00 09:25 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	505.1
Stddev	5.1
%RSD	1.016
#1	499.4
#2	503.4
#3	505.9
#4	511.6

500130

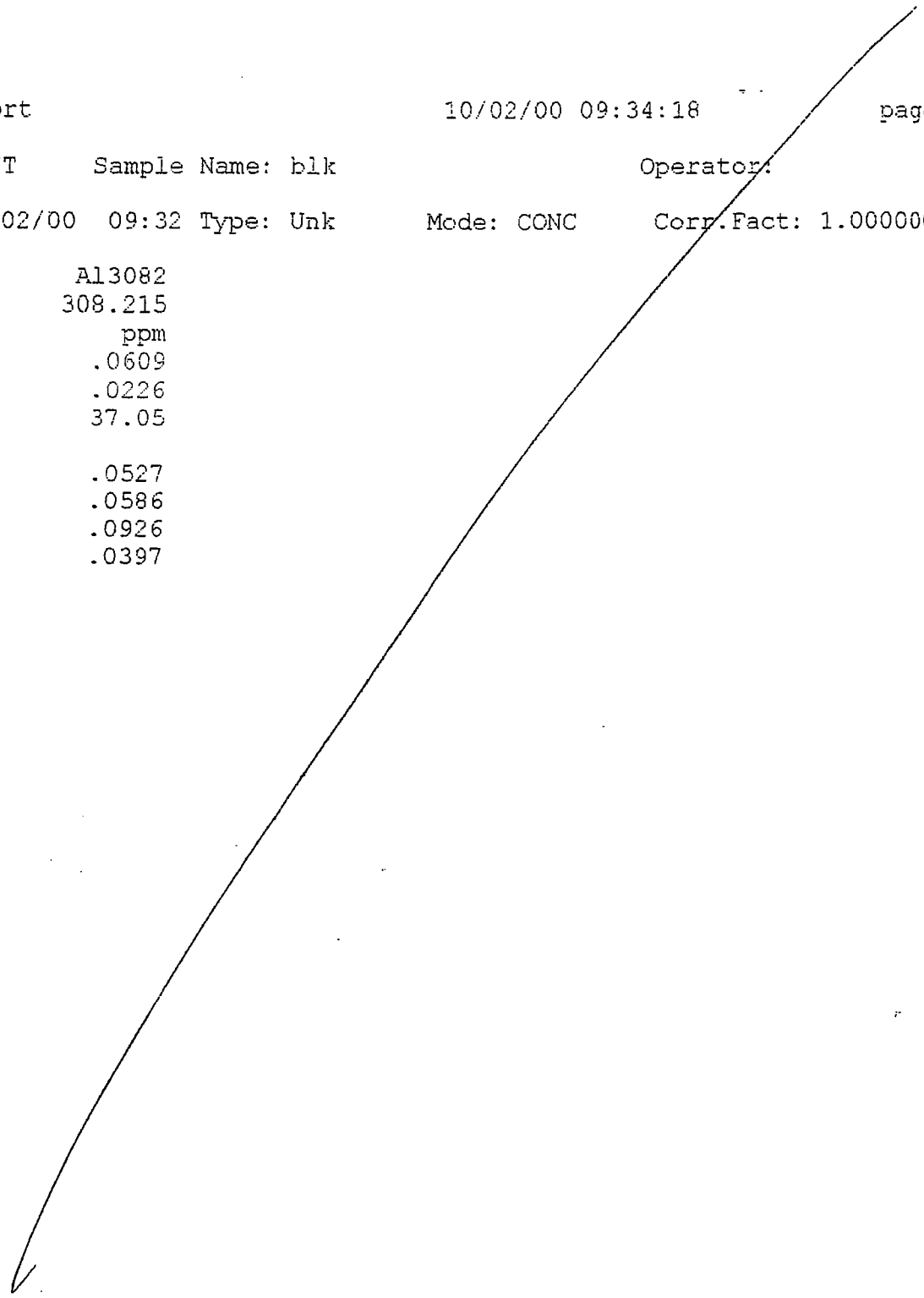
Method: ALTEST Sample Name: x nls 3 Operator:
Comment:
Run Time: 10/02/00 09:30 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	10.71
Stddev	.15
%RSD	1.370
#1	10.82
#2	10.52
#3	10.66
#4	10.83

Analysis Report 10/02/00 09:34:18 page 1

Method: ALTEST Sample Name: blk Operator:
Comment:
Run Time: 10/02/00 09:32 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	.0609
Stddev	.0226
%RSD	37.05
#1	.0527
#2	.0586
#3	.0926
#4	.0397



500132

Analysis Report 10/02/00 09:37:53 page 1

Method: ALTEST Sample Name: blk Operator:
Comment:
Run Time: 10/02/00 09:36 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	-.0150
Stddev	.0216
%RSD	143.7
#1	-.0047
#2	-.0022
#3	-.0059
#4	-.0473

500133

Analysis Report 10/02/00 10:04:58 page 1

Method: ALTEST Sample Name: 238341 Operator:
Comment:
Run Time: 10/02/00 10:03 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	91.73
Stddev	1.04
%RSD	1.132
#1	92.44
#2	90.48
#3	91.28
#4	92.71

500134

Method: ALTEST Sample Name: 238341 InstrSpike Operator:
Comment:
Run Time: 10/02/00 10:13 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	93.58
Stddev	.91
%RSD	.9707
#1	93.32
#2	92.69
#3	93.47
#4	94.84

Method: ALTEST Sample Name: 238341 InstruSpkDup Operator:
Comment:
Run Time: 10/02/00 10:17 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	93.09
Stddev	1.14
%RSD	1.228
#1	93.71
#2	94.08
#3	91.49
#4	93.06

Method: ALTEST Sample Name: 238342 Operator:
Comment:
Run Time: 10/02/00 10:21 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	86.76
Stddev	1.01
%RSD	1.166
#1	85.90
#2	87.97
#3	85.94
#4	87.21

Method: ALTEST Sample Name: 238404 Operator:
Comment:
Run Time: 10/02/00 10:46 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	128.0
Stddev	2.3
%RSD	1.760
#1	130.8
#2	128.2
#3	127.6
#4	125.3

Method: ALTEST Sample Name: 238404 InstrSpike Operator:
Comment:
Run Time: 10/02/00 10:49 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	108.8
Stddev	3.1
%RSD	2.841
#1	112.4
#2	110.1
#3	107.0
#4	105.5

Method: ALTEST Sample Name: 238404 InstruSpkDup Operator:
Comment:
Run Time: 10/02/00 10:53 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	112.1
Stddev	1.0
%RSD	.8698
#1	112.5
#2	112.7
#3	112.4
#4	110.6

500140

Method: ALTEST Sample Name: 238404 DigSpike Operator:
Comment:
Run Time: 10/02/00 10:57 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	104.6
Stddev	1.2
%RSD	1.188
#1	104.4
#2	104.7
#3	103.1
#4	106.1

Method: ALTEST Sample Name: 238404 DigSpkDup Operator:
Comment:
Run Time: 10/02/00 11:00 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	118.2
Stddev	1.8
%RSD	1.492
#1	119.4
#2	117.8
#3	115.8
#4	119.6

Analysis Report 10/02/00 11:09:24 page 1

Method: ALTEST Sample Name: Blank Operator:
Comment:
Run Time: 10/02/00 11:07 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	.0103
Stddev	.0153
%RSD	148.3
#1	.0135
#2	.0297
#3	-.0063
#4	.0042

ok

500143

Analysis Report 10/02/00 11:13:00 page 1

Method: ALTEST Sample Name: chkstd Operator:
Comment:
Run Time: 10/02/00 11:11 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	10.36
Stddev	.09
%RSD	.8712

OK

#1	10.34
#2	10.40
#3	10.25
#4	10.46

500144

Analysis Report 10/02/00 11:27:24 page 1

Method: ALTEST Sample Name: PrepBlk Operator:
Comment:
Run Time: 10/02/00 11:25 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	.0692
Stddev	.0062
%RSD	8.930
#1	.0771
#2	.0637
#3	.0710
#4	.0649

39

500145

Analysis Report 10/02/00 11:31:00 page 1

Method: ALTEST Sample Name: LFB Operator:
Comment:
Run Time: 10/02/00 11:29 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	10.43
Stddev	.13
%RSD	1.267
#1	10.39
#2	10.33
#3	10.38
#4	10.63

39

500146

Analysis Report 10/02/00 11:34:37 page 1

Method: ALTEST Sample Name: PrepBlk Operator:
Comment:
Run Time: 10/02/00 11:33 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	.0127
Stddev	.0094
%RSD	74.45
#1	.0181
#2	.0229
#3	.0025
#4	.0073

47

500147

Analysis Report 10/02/00 11:38:14 page 1

Method: ALTEST Sample Name: LFB Operator:
Comment:
Run Time: 10/02/00 11:36 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	9.560
Stddev	.197
%RSD	2.061
#1	9.302
#2	9.509
#3	9.723
#4	9.704

47

500148

Analysis Report 10/02/00 11:41:51 page 1

Method: ALTEST Sample Name: INF Operator:
Comment:
Run Time: 10/02/00 11:40 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	487.6
Stddev	5.2
%RSD	1.072
#1	482.0
#2	489.2
#3	485.0
#4	494.0

500149

Method: ALTEST Sample Name: ICS Operator:
Comment:
Run Time: 10/02/00 11:43 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	506.9
Stddev	4.1
%RSD	.8151
#1	502.8
#2	504.0
#3	510.9
#4	510.1

500150

Analysis Report 10/02/00 12:28:14 page 1

Method: ALTEST Sample Name: Blank Operator:
Comment:
Run Time: 10/02/00 12:26 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	-.0027
Stddev	.0110
%RSD	408.1
#1	.0000
#2	.0018
#3	.0061
#4	-.0187

500151

Analysis Report 10/02/00 12:31:51 page 1

Method: ALTEST Sample Name: chkstd Operator:
Comment:
Run Time: 10/02/00 12:30 Type: Unk Mode: CONC Corr.Fact: 1.000000

Elem	Al3082
Line	308.215
Units	ppm
Avg	10.69
Stddev	.22
%RSD	2.094
#1	10.41
#2	10.73
#3	10.67
#4	10.95

500152

NORTHERN LAKE SERVICE, INC.

ATTACHMENT 7

LEVEL 4 - QUALITY CONTROL DATA PACKAGE EXAMPLES

AA FURNACE METALS - METHOD 7740 – Selenium (Soil)

- NLS AA Furnace QC Data Forms for Metals Analysis of Soil
- NLS Analytical Bench Sheets for AA Furnace Metals
- NLS AA Furnace Metals Instrument Calibration / QC / Analysis Printouts

FORM I INORGANIC ANALYSIS DATA SHEET

Lab Name: Northern Lake Service Inc

SDG No.:(project) 56024, 56099, 56057

Lab Sample I.D.: See final report

Matrix (soil/water): (S) W

Date Received: See final report

% Solids See final report if applicable

Date analyzed: 9-13-2000

FORM 2A INITIAL AND CONTINUING CALIBRATION VERIFICATION

Initial Calibration Source: CPI XNLS-2 Lot# OCM031 Continuing Calibration Source: CPI XNLS-2 Lot# OCM031

Concentration Units: ug/L

Analyte	Initial Calibration			Continuing Calibration					
	True	Found	%R(1)	True	Found	%R(1)	Found	%R(1)	M
Selenium	25.0	25.08	100.3	25.0	1 24.55	98.2	2 26.81	107.2	EPA 7740
					3		4		EPA 7740

FORM 2B LOD STANDARD FOR AA AND ICP not applicable

FORM 3 BLANKS

Preparation Blank Matrix (soil/water): (S) W Preparation Blank Concentration Units (ug/L)

Analyte	Initial Calib. Blank (ug/L)	Q	Continuing Calibration Blank (ug/L)						Preparation Blank		
			CCB #	Q	CCB #	Q	CCB #	Q	Batch #	R	Q
Selenium	ND		1 ND		2 ND		3		35	ND	
			4		5		6				

FORM 4 ICP INTERFERENCE CHECK SAMPLE not applicable

FORM 5A SPIKE DUPLICATE

Matrix (soil/water): (S) W Level: _____ Concentration Units (ug/L)

Analyte	Sample Number	Spike Sample Result (SSR)	Q	Sample Result (SR)	Q	Spike Added (SA)	%R	Q	Method
Selenium instr	238370	23.77		ND		25.0	95.1		EPA 7740
Selenium dig						25.0			EPA 7740
Selenium instr	238386	25.25		ND		25.0	101.0		EPA 7740
Selenium dig						25.0			EPA 7740
Selenium instr						25.0			EPA 7740
Selenium dig						25.0			EPA 7740

FORM 7 LABORATORY CONTROL SAMPLE (LFB)

Aqueous LCS Source: CPI XNLS-2 Lot# OCM031

Analyte	Aqueous (ug/L)				Solid (ug/L)			
	True	Found	%R	Batch #	TRUE	Found	%R	Batch #
Selenium	25.0				12.5	10.70	85.6	35
Selenium	25.0				12.5			
Selenium	25.0				12.5			

FORM 9 ICP SERIAL DILUTIONS not applicable

FORM 10 METHOD DETECTION LIMITS

INSTR ID Number: PE 4100 ZL Date: 1/27/00

Analyte	Wave length (nm)	Back-ground	LOD (ug/L)	LOQ (ug/L)	Method
Selenium	196.0	Zeeman	1.7	6.0	EPA 7740

FORM 11A ICP INTERELEMENT CORRECTION FACTORS (ANNUALLY) not applicable

FORM 11B ICP INTERELEMENT CORRECTION FACTORS (ANNUALLY) not applicable

FORM 12 ICP LINEAR RANGES not applicable

500375

3520 -- Selenium, tot. as Se by furnace AAS

PRINTED: 09/12/00 13:36:00

TYPE: Metal (F) ID: 141

DATE 9-13-2000
ANALYST
CHECKED
ENTERED
SPK CON
VER DATE
STD DATE

LOD: (NON) 1.6 ug/L 27-JAN-00
LOQ: (NON) 6.1 ug/L 27-JAN-00
LOD: (DIG) 1.7 ug/L 27-JAN-00
LOQ: (DIG) 6.0 ug/L 27-JAN-00

HOLDTIME: 21 days
PAL: 10 ES: 50

METHODS: _____ SOP#
_____ EPA EPA 270.2
_____ SW846 SW846 7740
_____ ASTM SM 3113B
_____ NLS

MH = 138 S 2

MX	TYPE	UNITS	LOVAL	HIVAL	UCL	MEAN	LCL	DATE
C	DUP	ug/l	0	3	20.00		.00	
C	DUP	ug/l	3	10	20.00		.00	
C	DUP	ug/l	10	100	11.88	3.71	-4.46	03-MAR-99
C	SPK	%	0	200	123.34	92.28	61.22	03-MAR-99
LCS	DUP	ug/l	0	3	20.00		.00	
LCS	DUP	ug/l	3	10	20.00		.00	
LCS	DUP	ug/l	10	100	20.00		.00	
LCS	SPK	%	0	200	120.00		80.00	
S	DUP	mg/kg	0	3	20.00		.00	
S	DUP	mg/kg	3	10	20.00		.00	
S	DUP	mg/kg	10	100	17.31	4.90	-7.51	23-JAN-96
S	SPK	%	0	200	122.63	94.40	66.18	23-JAN-96
SC	DUP	ug/l	0	3	20.00		.00	
SC	DUP	ug/l	3	10	20.00		.00	
SC	DUP	ug/l	10	100	20.00		.00	
SC	SPK	%	0	200	120.00		80.00	
W	DUP	ug/l	0	3	20.00		.00	
W	DUP	ug/l	3	10	20.00		.00	
W	DUP	ug/l	10	100	21.80	5.03	-11.74	03-MAR-99
W	SPK	%	0	200	127.38	85.07	42.76	03-MAR-99

BLANK (BLK) - CHECKSTANDARD (CHK) - DUPLICATE (DUP) - RECOVERY (SPK) DATA

SAMPLE#	MTRX	TYPE	VALUE1	VALUE2	UNITS	WHO	DATE
238220	S	Spk	95.1		%	M	9-13-2000
238220	S	Dup	23.77	23.02	ug/L		
238386	S	Spk	104.0		%		
238386	S	Dup	25.25	26.14	ug/L		

3520 -- Selenium, tot. as Se by furnace AAS PAGE: 2

PRINTED: 09/12/00 13:36:00 DATE & TIME ANALYZED 9-13-2000

DEFAULT UNITS ug/L <1.7/33.77/95.17 <1.7/32.02/92.17

<1.7
238270 -- 56024 1 ~~20~~ 35 2 <0.89 mg/kg DWB QC x
[redacted] MAX: 14-SEP-00 – Soil, PGP00B003 – MTX= SO – 19 d-old 19 d-hse 14:15

1.99
238341 -- 56051 3 35 2.7 mg/kg DWB (2.3) QC x
[redacted] MAX: 14-SEP-00 – Soil, SD208006.5 – MTX= SO – 19 d-old 18 d-hse 09:17

<1.7
238342 -- 56051 4 35 <2.2 mg/kg DWB QC x
[redacted] MAX: 14-SEP-00 – Soil, SD208007.5 – MTX= SO – 19 d-old 18 d-hse 09:42

<1.7
238343 -- 56051 5 35SD <1.3 mg/kg DWB QC x
[redacted] MAX: 14-SEP-00 – Soil, SD208008.5 MS/ – MTX= SO – 19 d-old 18 d-hse 10:05

<1.7
238354 -- 56051 6 35 <3.7 mg/kg DWB QC x
[redacted] MAX: 14-SEP-00 – Soil, SD209006 – MTX= SO – 19 d-old 18 d-hse 15:36

<1.7
238355 -- 56051 7 35 <0.03 mg/kg WWB QC x
[redacted] MAX: 14-SEP-00 – EB016 – MTX= MS – 19 d-old 18 d-hse 16:17

<1.7
238356 -- 56051 8 35 <1.2 mg/kg DWB QC x
[redacted] MAX: 14-SEP-00 – Soil, SB209008.5 – MTX= SO – 19 d-old 18 d-hse 16:20

<1.7
238357 -- 56051 9 35 <1.1 mg/kg DWB QC x
[redacted] MAX: 14-SEP-00 – Soil, SB209008.5/D – MTX= SO – 19 d-old 18 d-hse 16:20

<1.7
238384 -- 56057 10 35 <0.98 mg/kg DWB QC x
[redacted] MAX: 15-SEP-00 – Soil, Swale 001001 – MTX= SO – 18 d-old 15 d-hse 07:30

<1.7
238385 -- 56057 11 35 <1.2 mg/kg DWB QC x
[redacted] MAX: 15-SEP-00 – Soil, Swale 002001 – MTX= SO – 18 d-old 15 d-hse 08:00

<1.7 <1.7/27.25/101.07 1.70/26.14/97.77
238386 -- 56057 12 35 13 <1.4 mg/kg DWB QC x
[redacted] MAX: 15-SEP-00 – Soil, Swale 003001 – MTX= SO – 18 d-old 15 d-hse 08:30

<1.7
238387 -- 56057 14 35 <0.03 mg/kg WWB QC x
[redacted] MAX: 15-SEP-00 – EB518 – MTX= MS – 18 d-old 15 d-hse 08:45

238526 -- 56081 ND
[redacted] MAX: 19-SEP-00 – FC082900A/B – MTX= MS – 14 d-old 13 d-hse 08:30

238551 -- 56088 ND
[redacted] MAX: 19-SEP-00 – NiaGro – MTX= MS – 14 d-old 13 d-hse 13:30 WW-10

238638 -- 56098 ND
[redacted] MAX: 19-SEP-00 – Digester #3 Cake – MTX= SL – 14 d-old 13 d-hse 11:30 WW-10

<1.7
238642 -- 56099 15 35 <1.6 <0.86 mg/kg DWB
[redacted] MAX: 18-SEP-00 – Soil, SD215000.5 – MTX= SO – 15 d-old 13 d-hse 11:31

<1.7
238643 -- 56099 16 35 <2.4 mg/kg DWB
[redacted] MAX: 18-SEP-00 – Soil, SD215002.5 – MTX= SO – 15 d-old 13 d-hse 11:56

1.72
238644 -- 56099 17 35 1.7 mg/kg DWB (1.7)
[redacted] MAX: 18-SEP-00 – Soil, SD215004.5 – MTX= SO – 15 d-old 13 d-hse 12:13

<1.7
238645 -- 56099 18 35SD <1.4 mg/kg DWB
[redacted] MAX: 18-SEP-00 – Soil, SB215006.5 MS/ – MTX= SO – 15 d-old 13 d-hse 16:45

500377

3520 -- Selenium, tot. as Se by furnace AAS PAGE: 3

PRINTED: 09/12/00 13:36:00 DATE & TIME ANALYZED 9-13-2000

DEFAULT UNITS ug/L

<1.7
238656 -- 56099 19 35 <0.03mg/kg WWB

[redacted] MAX: 18-SEP-00 -- EB018 -- MTX= MS -- 15 d-old 13 d-hse 16:33

[redacted] 14 d-old 11 d-hse 13:30

[redacted] 14 d-old 11 d-hse 11:30

[redacted] 14 d-old 11 d-hse 09:30

[redacted] 15 d-old 11 d-hse 06:30

[redacted] 15 d-old 11 d-hse 07:30

[redacted] 14 d-old 11 d-hse 00:00

[redacted] 13 d-old 11 d-hse 00:00

[redacted] 7 d-old 6 d-hse 10:00

[redacted] 7 d-old 6 d-hse 14:10 WW-10

[redacted] 6 d-old 5 d-hse 14:55

[redacted] 6 d-old 5 d-hse 14:00

[redacted] 5 d-old 5 d-hse 07:45

[redacted] 5 d-old 4 d-hse 15:46

[redacted] 12 d-old 4 d-hse 00:00

[redacted] 4 d-old 1 d-hse 07:15 QC X

[redacted] 4 d-old 1 d-hse 07:40 QC X

[redacted] 4 d-old 1 d-hse 07:50 QC X

[redacted] 4 d-old 1 d-hse 08:27 QC X

20 LFB 435" 85.6%

21 Blank 113" 4.7

500378

Element: Se Seq. No.: 68 AS Loc.: 36 Date: 09/13/2000
Sample ID: Calib Blank
µL dispensed: 10 from 36, 5 from 38, 20 from 36

Repl #	SampleConc	StndConc	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1			0.0016	0.0016	0.0198	0.0398	0.0773	06:21:18	No
2			0.0044	0.0044	0.0177	0.0385	0.0862	06:24:15	No
Mean:			0.0030						
SD :			0.0020						
%RSD:			67.1						

Auto-zero performed.

Element: Se Seq. No.: 69 AS Loc.: 37 Date: 09/13/2000
Sample ID: 12.5 ug/L
µL dispensed: 25 from 36, 5 from 38, 5 from 37

Repl #	SampleConc	StndConc	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1			0.0297	0.0326	0.0769	0.0467	0.0918	06:27:09	No
2			0.0301	0.0331	0.0799	0.0460	0.0838	06:30:05	No
Mean:			0.0299						
SD :			0.0003						
%RSD:			1.1						

[Se] Standard number 1 applied. [12.50]
Correlation Coefficient: 1.00000 Slope: 0.00239
Intercept : 0.00000

Element: Se Seq. No.: 70 AS Loc.: 37 Date: 09/13/2000
Sample ID: 25.0 ug/L
µL dispensed: 20 from 36, 5 from 38, 10 from 37

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1			0.0597	0.0627	0.1290	0.0498	0.0904	06:33:02	No
2			0.0635	0.0665	0.1338	0.0494	0.0956	06:35:59	No
Mean:			0.0616						
SD :			0.0027						
%RSD:			4.4						

[Se] Standard number 2 applied. [25.00]
Correlation Coefficient: 0.99986 Slope: 0.00246
Intercept : -0.00030

Element: Se Seq. No.: 71 AS Loc.: 37 Date: 09/13/2000
Sample ID: 50.0 ug/L
µL dispensed: 10 from 36, 5 from 38, 20 from 37

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1			0.1257	0.1287	0.2487	0.0623	0.1037	06:38:56	No
2			0.1194	0.1224	0.2536	0.0623	0.1015	06:41:53	No
Mean:			0.1226						
SD :			0.0045						
%RSD:			3.6						

[Se] Standard number 3 applied. [50.00]
Correlation Coefficient: 0.99997 Slope: 0.00246
Intercept : -0.00023

500379

Calibration data for Se

Standard ID	Mean Signal (Pk Area)	Entered Concentration (µg/L)	Calculated Concentration (µg/L)	Standard Deviation	%RSD
Calib Blank	0.0030	--	-----	----	----
12.5 ug/L	0.0299	12.50	12.26	0.000	1.1
25.0 ug/L	0.0616	25.00	25.17	0.003	4.4
50.0 ug/L	0.1226	50.00	49.98	0.004	3.6

Correlation Coefficient: 0.99997 Slope: 0.00246 Intercept: -0.0002

Element: Se Seq. No.: 72 AS Loc.: 39 Date: 09/13/2000
Sample ID: xnls#2
µL dispensed: 10 from 36, 5 from 38, 20 from 39

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	25.41	25.41	0.0622	0.0652	0.1356	0.0482	0.0921	06:44:51	No
2	24.75	24.75	0.0606	0.0635	0.1266	0.0478	0.0967	06:47:49	No
Mean:	25.08	25.08	0.0614						
SD :	0.469	0.469	0.0012						
%RSD:	1.9	1.9	1.9						

QC value within specified limits.

Element: Se Seq. No.: 73 AS Loc.: 36 Date: 09/13/2000
Sample ID: blank
µL dispensed: 10 from 36, 5 from 38, 20 from 36

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	-0.39	-0.39	-0.0012	0.0018	0.0196	0.0343	0.0756	06:50:51	No
2	-0.32	-0.32	-0.0010	0.0019	0.0158	0.0304	0.0670	06:53:47	No
Mean:	-0.35	-0.35	-0.0011						
SD :	0.047	0.047	0.0001						
%RSD:	13.3	13.3	10.5						

QC value within specified limits.

Element: Se Seq. No.: 74 AS Loc.: 1 Date: 09/13/2000
Sample ID: 238270
µL dispensed: 10 from 36, 5 from 38, 20 from 1

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	0.79	0.79	0.0017	0.0047	0.0383	0.2415	0.2013	06:56:43	No
2	-1.06	-1.06	-0.0028	0.0001	0.0343	0.2496	0.1953	06:59:40	No
Mean:	-0.14	-0.14	-0.0006						
SD :	1.306	1.306	0.0032						
%RSD:	961.3	961.3	566.3						

Element: Se Seq. No.: 75 AS Loc.: 1 Date: 09/13/2000
Sample ID: 238270
µL dispensed: 5 from 38, 10 from 37, 20 from 1

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	23.88	23.88	0.0584	0.0614	0.1739	0.2758	0.2165	07:02:37	No
2	23.66	23.66	0.0579	0.0608	0.1577	0.2778	0.2091	07:05:33	No

500380

Mean:	23.77	23.77	0.0582
SD :	0.157	0.157	0.0004
%RSD:	0.7	0.7	0.7

Recovery for Se = 95.1 % within 80 % to 120 %

Element: Se Seq. No.: 76 AS Loc.: 2 Date: 09/13/2000
Sample ID: 238270 dup
µL dispensed: 10 from 36, 5 from 38, 20 from 2

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	-0.03	-0.03	-0.0003	0.0027	0.0402	0.2419	0.1782	07:08:29	No
2	1.24	1.24	0.0028	0.0058	0.0384	0.2466	0.1840	07:11:26	No
Mean:	0.60	0.60	0.0013						
SD :	0.893	0.893	0.0022						
%RSD:	147.6	147.6	175.1						

Element: Se Seq. No.: 77 AS Loc.: 2 Date: 09/13/2000
Sample ID: 238270 dup
µL dispensed: 5 from 38, 10 from 37, 20 from 2

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	21.98	21.98	0.0538	0.0567	0.1572	0.2708	0.2037	07:14:21	No
2	24.06	24.06	0.0589	0.0619	0.1719	0.2672	0.1993	07:17:16	No
Mean:	23.02	23.02	0.0563						
SD :	1.478	1.478	0.0036						
%RSD:	6.4	6.4	6.4						

Recovery for Se = 92.1 % within 80 % to 120 %

Element: Se Seq. No.: 78 AS Loc.: 3 Date: 09/13/2000
Sample ID: 238341
µL dispensed: 10 from 36, 5 from 38, 20 from 3

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	2.73	2.73	0.0065	0.0094	0.0238	0.0825	0.0706	07:20:12	No
2	1.26	1.26	0.0029	0.0058	0.0224	0.0698	0.0679	07:23:09	No
Mean:	1.99	1.99	0.0047						
SD :	1.045	1.045	0.0026						
%RSD:	52.4	52.4	55.0						

Element: Se Seq. No.: 79 AS Loc.: 4 Date: 09/13/2000
Sample ID: 238342
µL dispensed: 10 from 36, 5 from 38, 20 from 4

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	1.26	1.26	0.0029	0.0058	0.0175	0.0651	0.0725	07:26:05	No
2	0.38	0.38	0.0007	0.0036	0.0209	0.0643	0.0729	07:29:01	No
Mean:	0.82	0.82	0.0018						
SD :	0.625	0.625	0.0015						
%RSD:	76.4	76.4	86.4						

Element: Se Seq. No.: 80 AS Loc.: 5 Date: 09/13/2000
Sample ID: 238343
µL dispensed: 10 from 36, 5 from 38, 20 from 5

500381

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	0.52	0.52	0.0010	0.0040	0.0321	0.1391	0.1091	07:31:57	No
2	0.78	0.78	0.0017	0.0046	0.0225	0.1426	0.1100	07:34:54	No
Mean:	0.65	0.65	0.0014						
SD :	0.187	0.187	0.0005						
%RSD:	28.7	28.7	33.5						

Element: Se Seq. No.: 81 AS Loc.: 6 Date: 09/13/2000
Sample ID: 238354
µL dispensed: 10 from 36, 5 from 38, 20 from 6

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	0.53	0.53	0.0011	0.0040	0.0233	0.0605	0.0637	07:37:49	No
2	1.10	1.10	0.0025	0.0054	0.0186	0.0603	0.0627	07:40:47	No
Mean:	0.82	0.82	0.0018						
SD :	0.406	0.406	0.0010						
%RSD:	49.7	49.7	56.2						

Element: Se Seq. No.: 82 AS Loc.: 7 Date: 09/13/2000
Sample ID: 238355
µL dispensed: 10 from 36, 5 from 38, 20 from 7

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	-0.59	-0.59	-0.0017	0.0013	0.0121	0.0233	0.0531	07:43:42	No
2	-0.65	-0.65	-0.0018	0.0011	0.0130	0.0270	0.0527	07:46:39	No
Mean:	-0.62	-0.62	-0.0018						
SD :	0.039	0.039	0.0001						
%RSD:	6.4	6.4	5.5						

Element: Se Seq. No.: 83 AS Loc.: 8 Date: 09/13/2000
Sample ID: 238356
µL dispensed: 10 from 36, 5 from 38, 20 from 8

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	0.48	0.48	0.0010	0.0039	0.0248	0.1526	0.1552	07:49:36	No
2	-0.72	-0.72	-0.0020	0.0010	0.0249	0.1534	0.1384	07:52:33	No
Mean:	-0.12	-0.12	-0.0005						
SD :	0.847	0.847	0.0021						
%RSD:	720.8	720.8	399.1						

Element: Se Seq. No.: 84 AS Loc.: 9 Date: 09/13/2000
Sample ID: 238357
µL dispensed: 10 from 36, 5 from 38, 20 from 9

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	1.04	1.04	0.0023	0.0053	0.0195	0.1524	0.1511	07:55:30	No
2	0.09	0.09	0.0000	0.0029	0.0233	0.1617	0.1558	07:58:28	No
Mean:	0.56	0.56	0.0012						
SD :	0.668	0.668	0.0016						
%RSD:	118.5	118.5	142.4						

Element: Se Seq. No.: 85 AS Loc.: 10 Date: 09/13/2000
Sample ID: 238384

500382

µL dispensed: 10 from 36, 5 from 38, 20 from 10

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	0.80	0.80	0.0017	0.0047	0.0285	0.1851	0.1592	08:01:25	No
2	0.34	0.34	0.0006	0.0036	0.0212	0.1904	0.1667	08:04:25	No
Mean:	0.57	0.57	0.0012						
SD :	0.320	0.320	0.0008						
%RSD:	56.1	56.1	67.3						

Element: Se Seq. No.: 86 AS Loc.: 11 Date: 09/13/2000
Sample ID: 238385
µL dispensed: 10 from 36, 5 from 38, 20 from 11

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	1.11	1.11	0.0025	0.0054	0.0275	0.2042	0.1789	08:07:23	No
2	0.68	0.68	0.0014	0.0044	0.0369	0.2081	0.1858	08:10:22	No
Mean:	0.89	0.89	0.0020						
SD :	0.302	0.302	0.0007						
%RSD:	33.8	33.8	37.8						

Element: Se Seq. No.: 87 AS Loc.: 39 Date: 09/13/2000
Sample ID: xnls#2
µL dispensed: 10 from 36, 5 from 38, 20 from 39

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	24.09	24.09	0.0590	0.0619	0.1243	0.0441	0.0702	08:13:22	No
2	25.01	25.01	0.0612	0.0642	0.1359	0.0426	0.0775	08:16:22	No
Mean:	24.55	24.55	0.0601						
SD :	0.647	0.647	0.0016						
%RSD:	2.6	2.6	2.6						

QC value within specified limits.

Element: Se Seq. No.: 88 AS Loc.: 36 Date: 09/13/2000
Sample ID: blank
µL dispensed: 10 from 36, 5 from 38, 20 from 36

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	0.47	0.47	0.0009	0.0039	0.0210	0.0253	0.0536	08:19:24	No
2	0.65	0.65	0.0014	0.0043	0.0147	0.0210	0.0598	08:22:20	No
Mean:	0.56	0.56	0.0011						
SD :	0.127	0.127	0.0003						
%RSD:	22.7	22.7	27.3						

QC value within specified limits.

Element: Se Seq. No.: 89 AS Loc.: 12 Date: 09/13/2000
Sample ID: 238386
µL dispensed: 10 from 36, 5 from 38, 20 from 12

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	1.37	1.37	0.0031	0.0061	0.0264	0.1547	0.1215	08:25:13	No
2	0.95	0.95	0.0021	0.0051	0.0223	0.1623	0.1094	08:28:12	No
Mean:	1.16	1.16	0.0026						
SD :	0.293	0.293	0.0007						
%RSD:	25.2	25.2	27.5						

500383

Element: Se Seq. No.: 90 AS Loc.: 12 Date: 09/13/2000
Sample ID: 238386
µL dispensed: 5 from 38, 10 from 37, 20 from 12

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	24.38	24.38	0.0597	0.0626	0.1787	0.1852	0.1110	08:31:08	No
2	26.12	26.12	0.0640	0.0669	0.1885	0.1871	0.1202	08:34:05	No
Mean:	25.25	25.25	0.0618						
SD :	1.235	1.235	0.0030						
%RSD:	4.9	4.9	4.9						

Recovery for Se = 101.0 % within 80 % to 120 %

Element: Se Seq. No.: 91 AS Loc.: 13 Date: 09/13/2000
Sample ID: 238386 dup
µL dispensed: 10 from 36, 5 from 38, 20 from 13

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	1.82	1.82	0.0042	0.0072	0.0339	0.1678	0.1119	08:37:04	No
2	1.59	1.59	0.0037	0.0066	0.0257	0.1644	0.1110	08:40:04	No
Mean:	1.70	1.70	0.0040						
SD :	0.166	0.166	0.0004						
%RSD:	9.7	9.7	10.3						

Element: Se Seq. No.: 92 AS Loc.: 13 Date: 09/13/2000
Sample ID: 238386 dup
µL dispensed: 5 from 38, 10 from 37, 20 from 13

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	25.91	25.91	0.0634	0.0664	0.1784	0.1893	0.1228	08:43:01	No
2	26.36	26.36	0.0645	0.0675	0.1836	0.1895	0.1258	08:45:59	No
Mean:	26.14	26.14	0.0640						
SD :	0.321	0.321	0.0008						
%RSD:	1.2	1.2	1.2						

Recovery for Se = 97.7 % within 80 % to 120 %

Element: Se Seq. No.: 93 AS Loc.: 14 Date: 09/13/2000
Sample ID: 238387
µL dispensed: 10 from 36, 5 from 38, 20 from 14

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	0.04	0.04	-0.0001	0.0028	0.0130	0.0282	0.0596	08:48:59	No
2	0.21	0.21	0.0003	0.0032	0.0126	0.0276	0.0649	08:51:57	No
Mean:	0.12	0.12	0.0001						
SD :	0.116	0.116	0.0003						
%RSD:	92.7	92.7	384.1						

Element: Se Seq. No.: 94 AS Loc.: 15 Date: 09/13/2000
Sample ID: 238642
µL dispensed: 10 from 36, 5 from 38, 20 from 15

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	1.86	1.86	0.0043	0.0073	0.0176	0.0663	0.0757	08:54:57	No
2	0.88	0.88	0.0019	0.0049	0.0180	0.0645	0.0705	08:57:56	No
Mean:	1.37	1.37	0.0031						
SD :	0.690	0.690	0.0017						

500384

%RSD: 50.3 50.3 54.1

Element: Se Seq. No.: 95 AS Loc.: 16 Date: 09/13/2000
Sample ID: 238643
µL dispensed: 10 from 36, 5 from 38, 20 from 16

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	-0.49	-0.49	-0.0014	0.0015	0.0137	0.0663	0.0736	09:00:56	No
2	0.24	0.24	0.0004	0.0033	0.0141	0.0662	0.0751	09:03:55	No
Mean:	-0.12	-0.12	-0.0005						
SD :	0.513	0.513	0.0013						
%RSD:	415.0	415.0	235.0						

Element: Se Seq. No.: 96 AS Loc.: 17 Date: 09/13/2000
Sample ID: 238644
µL dispensed: 10 from 36, 5 from 38, 20 from 17

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	1.85	1.85	0.0043	0.0073	0.0159	0.0847	0.0825	09:06:55	No
2	1.59	1.59	0.0037	0.0066	0.0185	0.0839	0.0823	09:09:54	No
Mean:	1.72	1.72	0.0040						
SD :	0.186	0.186	0.0005						
%RSD:	10.8	10.8	11.4						

Element: Se Seq. No.: 97 AS Loc.: 18 Date: 09/13/2000
Sample ID: 238645
µL dispensed: 10 from 36, 5 from 38, 20 from 18

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	0.98	0.98	0.0022	0.0051	0.0220	0.0991	0.1207	09:12:55	No
2	-0.04	-0.04	-0.0003	0.0026	0.0173	0.1067	0.1089	09:15:54	No
Mean:	0.47	0.47	0.0009						
SD :	0.721	0.721	0.0018						
%RSD:	152.3	152.3	190.3						

Element: Se Seq. No.: 98 AS Loc.: 19 Date: 09/13/2000
Sample ID: 238656
µL dispensed: 10 from 36, 5 from 38, 20 from 19

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	-0.90	-0.90	-0.0024	0.0005	0.0142	0.0284	0.0636	09:18:53	No
2	-0.55	-0.55	-0.0016	0.0014	0.0157	0.0304	0.0651	09:21:52	No
Mean:	-0.73	-0.73	-0.0020						
SD :	0.248	0.248	0.0006						
%RSD:	34.1	34.1	30.1						

Element: Se Seq. No.: 99 AS Loc.: 20 Date: 09/13/2000
Sample ID: lfb 35
µL dispensed: 10 from 36, 5 from 38, 20 from 20

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	11.00	11.00	0.0268	0.0298	0.0630	0.0318	0.0696	09:24:50	No
2	10.40	10.40	0.0253	0.0283	0.0683	0.0341	0.0667	09:27:46	No

500385

Mean:	10.70	10.70	0.0261
SD :	0.422	0.422	0.0010
%RSD:	3.9	3.9	4.0

Element: Se Seq. No.: 100 AS Loc.: 21 Date: 09/13/2000
Sample ID: blank 35
µL dispensed: 10 from 36, 5 from 38, 20 from 21

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	0.76	0.76	0.0016	0.0046	0.0143	0.0263	0.0601	09:30:43	No
2	-0.52	-0.52	-0.0015	0.0014	0.0132	0.0261	0.0570	09:33:39	No
Mean:	0.12	0.12	0.0001						
SD :	0.903	0.903	0.0022						
%RSD:	771.7	771.7	4045.1						

Element: Se Seq. No.: 101 AS Loc.: 39 Date: 09/13/2000
Sample ID: xnls#2
µL dispensed: 10 from 36, 5 from 38, 20 from 39

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	25.69	25.69	0.0629	0.0658	0.1303	0.0435	0.0975	09:36:34	No
2	27.94	27.94	0.0684	0.0714	0.1369	0.0470	0.1014	09:39:33	No
Mean:	26.81	26.81	0.0656						
SD :	1.591	1.591	0.0039						
%RSD:	5.9	5.9	6.0						

QC value within specified limits.

Element: Se Seq. No.: 102 AS Loc.: 36 Date: 09/13/2000
Sample ID: blank
µL dispensed: 10 from 36, 5 from 38, 20 from 36

Repl #	SampleConc µg/L	StndConc µg/L	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	-0.23	-0.23	-0.0008	0.0021	0.0176	0.0400	0.0784	09:42:35	No
2	-0.47	-0.47	-0.0014	0.0016	0.0155	0.0346	0.0901	09:45:32	No
Mean:	-0.35	-0.35	-0.0011						
SD :	0.169	0.169	0.0004						
%RSD:	48.0	48.0	37.8						

QC value within specified limits.

500386

NORTHERN LAKE SERVICE, INC.

ATTACHMENT 8

LEVEL 4 - QUALITY CONTROL DATA PACKAGE EXAMPLES

AA Cold Vapor - METHOD 7470 – Mercury (Soil)

- NLS AA Cold Vapor QC Data Forms for Mercury Analysis of Soil
- NLS Analytical Bench Sheets for AA Cold Vapor Mercury Analysis
- NLS AA Cold Vapor Mercury - Instrument Calibration / QC / Analysis Data Sheets
- NLS – Anomaly / Incident Report form – describing an Anomaly
- NLS AA Cold Vapor Mercury - Instrument Calibration / QC / Analysis Chart

FORM I INORGANIC ANALYSIS DATA SHEET

Lab Name: Northern Lake Service, Inc

SDG No.:(project) 63447, 63579

Lab Sample I.D.: See final report

Matrix (soil)

Date Received: See final report

% Solids See final report

Date analyzed: 11-14-01

FORM 2A INITIAL AND CONTINUING CALIBRATION VERIFICATION

Correlation Coefficient: .9989 Slope: .2527 Y-intercept: -.028

Initial Calibration Source: Spex Hg#8-32HG-X Continuing Calibration Source: Spex Hg#8-17Hg

Concentration Units: ug/L

Analyte	Initial Calibration			Continuing Calibration					
	True	Found	%R(1)	True	Found	%R(1)	Found	%R(1)	M
Mercury	0.50	.503	101%	0.50	1 .483	97%	2 .499	100%	7470
					3 .495	99%	4		7470

FORM 2B LOD STANDARD FOR AA AND ICP not applicable

FORM 3 BLANKS

Preparation Blank Matrix (soil): Preparation Blank Concentration Units (ug/L)

Analyte	Initial Calib. Blank (ug/L)	Q	Continuing Calibration Blank (ug/L)						Preparation Blank	
			CCB #	Q	CCB #	Q	CCB #	Q	R	Q
Mercury	ND		1 ND		2 ND		3 ND		ND	

FORM 4 ICP INTERFERENCE CHECK SAMPLE not applicable

FORM 5A SPIKE DUPLICATE

Matrix (soil): Level: 0.50 ug/L Concentration Units (ug/L)

Analyte	Sample Number	Spike Sample Result (SSR)	Q	Sample Result (SR)	Q	Spike Added (SA)	%R	Q	Method
Mercury dig	268701	.634		.115		0.50	104%		7470
Mercury dig	268710	.658		.242		0.50	83%		7470
Mercury dig	269224	<.02		.499		0.50	100%		7470
Mercury dig						0.50			7470

FORM 7 LABORATORY CONTROL SAMPLE (LFB)

Aqueous LCS Source: Spex Hg#8-17Hg

Analyte	Aqueous (ug/L)		
	True	Found	%R
Mercury	0.50	.519	104%

FORM 9 ICP SERIAL DILUTIONS not applicable

FORM 10 METHOD DETECTION LIMITS

INSTR ID Number: Varian AA1475 Date: 9/21/01

Analyte	Wave length (nm)	Back-ground	LOD (ug/L)	LOQ (ug/L)	Method
Mercury	253.7	NA	0.022	0.078	7470

FORM 11A ICP INTERELEMENT CORRECTION FACTORS (ANNUALLY) not applicable

FORM 11B ICP INTERELEMENT CORRECTION FACTORS (ANNUALLY) not applicable

FORM 12 ICP LINEAR RANGES not applicable

2610 - Mercury, total as Hg in solids - 2610 [illegible] 11/12/01 08:38 Page 1 of 3

Type: Metal (CV) - S2 - 1378 Holdtime: 28 days Links: PAL : ES : MCL

Notes	
Original	Analyzed: 11-14-01 Analyst SAC Checked ____ Entered ____

#	INSTRUMENT	LOD	LOQ		DLOD	DLOQ		EPA	SW	SM
1	Hg,tot CVAA,soil/slg	9-21-01 ~~07/17/00~~ TRP	11-27-01	mg/Kg DWB	~~0.0065~~ 0.066 07/17/00	~~0.317~~ 0.33 TRP 11-27-01	mg/Kg DWB	EPA 245.1	SW846 7470A	SM 3112B

MX	TYPE	UNITS	LOVAL	HIVAL	UCL	UWL	MEAN	LWL	LCL	DATE
W	DUP	ug/l	0	20	53.77	38.92	9.22	-20.48	-35.33	08/11/01
W	DUP	ug/l	20	50	20.00				0.00	
W	DUP	ug/l	50	100	20.00				0.00	
W	SPK	%	0	200	172.62	146.43	94.03	41.64	15.44	08/11/01

BLANK (BLK) - CHECKSTANDARD (CHK) - DUPLICATE (DUP) - RECOVERY (SPK) DATA

SAMPLE#	MTX	TYPE	VALUE1	VALUE2	UNITS	WHO	DATE
268701	W	Dp	.634	.721	ppb	SAC	11-14-01
11	↓	Spk	104		%R	↓	↓
268710			.658	.662			
11			83				
269224			.499	.527			
11		↓	100		↓		

End

Date & Time Analyzed | Default Units mg/Kg DWB | Res mg/kg

MDL | LOQ

[redacted] INC%SOL | Sn X

[redacted] X:11/05/01 - MTX:MS - 34.9d-old 33d-hse 11:15

2.7%SOL

[redacted] dge - MAX:11/19/01 - MTX:SL - 20.9d-old 20d-hse 10:11 | WW-10

1.2%SOL

[redacted] AX:11/21/01 - MTX:SL - 18.9d-old 19d-hse 09:45

42.4%SOL

[redacted] nal Sludge - MAX:11/16/01 - MTX:SL - 24d-old 18d-hse 08:00

3.4%SOL

[redacted] igested Sludge - MAX:11/27/01 - MTX:SL - 13.1d-old 13d-hse 07:00

5.7%SOL

[redacted] .T.P. Raw Sludge - MAX:11/26/01 - MTX:SL - 13.8d-old 12d-hse 13:00

Sample		Handwritten		Level	%SOL	MDL	LOQ	Res
268700 -- 63447	1	.597g	.099	Q Level 4	33.5%SOL	.10 SAC	.39 .32 SAC	.49

[redacted] - SB483000.5, 30166-70 - MAX:11/27/01 - MTX:SO - 13d-old 12d-hse 09:15

268701 -- 63447	2	.529g .115 (3) .634	1043 (4) .724	Q Level 4	37.8%SOL	.10 .11	.32 .39	.57

[redacted] - SB482002.5, 30171-75 ... MAX:11/27/01 - MTX:SO ... 12d-hse 09:30

268702 -- 63447	5	.966g	.096 mg	Q Level 4	20.7%SOL	.10 .11	.32 .39	.48

[redacted] - SB485000.5, 30231-35 - MAX:11/27/01 - MTX:SO - 12.9d-old 12d-hse 10:30

268703 -- 63447	6	.649g	.230 mg	Q Level 4	30.8%SOL	.10 .11	.32 .39	1.1

[redacted] - SB485001.2, 30236-40 - MAX:11/27/01 - MTX:SO - 12.9d-old 12d-hse 10:45

268704 -- 63447	7	1.02g	.111 mg	Q Level 4	19.7%SOL	.10 .11	.32 .39	.5

[redacted] - SB484000.5, 30196-200 - MAX:11/27/01 - MTX:SO - 12.9d-old 12d-hse 10:30

268705 -- 63447	8	.583g	.088 mg	Q Level 4	34.3%SOL	.10 .11	.32 .39	.44

[redacted] - SB484002.5, 30201-05 - MAX:11/27/01 - MTX:SO - 12.9d-old 12d-hse 11:45

268706 -- 63447	9	.588g	.107 mg	Q Level 4	34%SOL	.10 .11	.32 .39	.5

[redacted] - SB484002.5/D, 30206-10 - MAX:11/27/01 - MTX:SO - 12.9d-old 12d-hse 11:45

268707 -- 63447	10	.499g	.183 mg	Q Level 4	40.1%SOL	.10 .11	.32 .39	.91

[redacted] - SB484004, 30211-15 - MAX:11/27/01 - MTX:SO - 12.8d-old 12d-hse 12:00

268708 -- 63447	11	.741g	.131 mg	Q Level 4	27%SOL	.10 .11	.32 .39	.48

[redacted] - SB486000.5, 30266-70 - MAX:11/27/01 - MTX:SO - 12.8d-old 12d-hse 13:15

268709 -- 63447	12	.683g	.262 mg	Q Level 4	29.3%SOL	.10 .11	.32 .39	1.3

[redacted] - SB486001.5, 30271-75 - MAX:11/27/01 - MTX:SO - 12.8d-old 12d-hse 13:30

268710 -- 63447	13	.660g .242 (14) .158	8350 (15) .662	Q Level 4	30.3%SOL	.11 .10	.32 .39	1.

[redacted] 5/D, 30281-85 - MAX:11/27/01 - MTX:SO - 12.8d-old 12d-hse 13:30

268711 -- 63447	16	.322g	.115	Q Level 4	62.1%SOL	.11 .10	.32 .39	.5

[redacted] - SB487000.5, 30301-05 - MAX:11/27/01 - MTX:SO - 12.8d-old 12d-hse 14:15

268712 -- 63447	17	.237g	<.02	Q Level 4	84.5%SOL	.11 .10	.32 .39	<

[redacted] - SB487001.2, 30306-10 - MAX:11/27/01 - MTX:SO - 12.7d-old 12d-hse 14:30

268713 -- 63447	18	100ml	.179	Q Level 4		SAC .10	SAC .32	Res

[redacted] - EB003, 30316, 33-35 - MAX:11/27/01 - MTX:SO - 12.8d-old 12d-hse 13:10

26.8%SOL SAC .10

[redacted] ludge - MAX:11/27/01 - MTX:SL - 13.3d-old 12d-hse 00:00

[redacted] :SO - 11.9d-old 11d-hse 09:52

500137

Date & Time Analyzed | Default Units mg/Kg DWB

				MDL	LOQ	Result mg/kg DW
269216 -- 63579 19	.255g	<.02	Q Level 4 QC 78	3%SOL .11	.32 .39	< .10 .11
[redacted] - SB488009 30058-64 - MAX:12/04/01 - MTX:SO - 6d-old 5d-hse 09:15						
269217 -- 63579 20	.248g	Reset (.464)	Q Level 4 QC 80	7%SOL .11 .10	.32 .39	(2.32)
[redacted] - SB489009, 30065-71 - MAX:12/04/01 - MTX:SO - 5.9d-old 5d-hse 10:00						
269218 -- 63579 21	.252g	Reset (.899)	Q Level 4 QC 79	4%SOL .11 .10	.32 .39	(4.49)
[redacted] - SB489009/D, 30072-78 - MAX:12/04/01 - MTX:SO - 5.9d-old 5d-hse 10:00						
269219 -- 63579 22	.272g	.056 mg	Q Level 4 QC 73	4%SOL .11 .10	.32 .39	.281
[redacted] - SB490088, 30079-85 - MAX:12/04/01 - MTX:SO - 5.9d-old 5d-hse 10:30						
269220 -- 63579 23	.237g	Reset (.373)	Q Level 4 QC 84	4%SOL .11 .10	.32 .39	(1.86)
[redacted] - SB491064, 30086-92 - MAX:12/04/01 - MTX:SO - 5.9d-old 5d-hse 11:05						
269221 -- 63579 24	.267g	.076 mg	Q Level 4 QC 74	8%SOL .11 .10	.32 .39	.381
[redacted] - SB492007, 30093-99 - MAX:12/04/01 - MTX:SO - 5.8d-old 5d-hse 13:15						
269222 -- 63579 25	.273g	<.02 mg	Level 4 QC 73	3%SOL .11 .10	.32 .39	< .10 .1 SAC
[redacted] - SB493007, 30100-06 - MAX:12/04/01 - MTX:SO - 5.8d-old 5d-hse 13:50						
269223 -- 63579 26	.231g	Reset	Q Level 4 QC 86	7%SOL .11 .10	.32 .39	
[redacted] - SB494003 30107-13 - MAX:12/04/01 - MTX:SO - 5.8d-old 5d-hse 14:15						
269224 -- 63579 27	.239g	<.02 (28) .499 (29) .527	Level 4 QC 83	7%SOL .11 .10	.32 .39	< .10 .11 SAC
[redacted] - SB495003 30114-20 MS/MSD - MAX:12/04/01 - MTX:SO - 5.8d-old 5d-hse 14:30						
269225 -- 63579 30	.249g	.056 mg	Q Level 4 QC 80	3%SOL .11 .10	.32 .39	.280
[redacted] - SB496002, 30121-27 - MAX:12/04/01 - MTX:SO - 5.7d-old 5d-hse 14:45						

Test Hg CV
Date 11-14-01
Analyst EMC

tot. ug Hg

Sample #	Abs. Conc.	Final Results	Sample #	Abs. Conc.	Final Results
Blk	.001		Blk	.001	<.02 ok
.05	.015	corr:	STD	.125	.483 = 97%
.10	.032	.9989	13	.064	.242
.20	.052	slope: .2527	(14)	.169	.658 = 83%
.50	.129	yint: .0008	(15)	.170	.662
1.00 Not used	.208		16	.032	.115
PB	.002	ok < .02	17	.006	<.02
LFM	.134	.519 = 104%	18	.048	.179 — Reset
VER	.130	.503 = 101%	19	.006	<.02
1	.028	.099	20	.120	.464 — Reset
(2)	.032	.115	21	.230	.899 — Reset
(3)	.163	.634 = 104%	22	.017	.056
(4)	.185	.721	23	.097	.373 — Reset
5	.027	.096	24	.022	.076
6	.061	.230	Blk	.001	<.02 ok
7	.031	.111	STD	.129	100% ok .499
8	.025	.088	25	.004	<.02
9	.030	.107	26	Blowoff	— — Reset
10	.049	.183	27	.005	<.02
11	.036	.131	(28)	.129	.499 = 100%
12	.069	.262	(29)	.136	.527

Test ~~HG~~ CV
Date 11-14-01
Analyst JMC

Sample #	Abs. Conc.	Final Results	Sample #	Abs. Conc.	Final Results
30	.017	.056 mg			
Blk	.002	ok			
Std	.128	.495 = 99%			

500140

NORTHERN LAKE SERVICE
CONTINUOUS QUALITY IMPROVEMENT
ANOMALY / INCIDENT REPORT
(Laboratory)

AI-209

Section 1: Initial Information (To be completed and distributed by originator)

Laboratory Area Affected: Metals (ICP) Reported by: Joe Cebe

Occurrence Date: 11-14-2001 Occurrence Time: AM

ANOMALY / INCIDENT ITEMS - Laboratory Areas

- ☐ Holding Time Exceeded
- ☐ Calibration Standard Exceed Limits
- ☐ Surrogates Exceed Limits
- ☐ Data Calculation Error
- ☐ Violation of Laboratory Policy
- ☐ Lab Security Violation
- ☐ Transcription Error
- ☐ Insufficient Sample Volume
- ☐ Laboratory Blank Contaminated
- ☐ Chain-of-Custody Violated
- ☐ Deviation from Standard Procedure
- ☐ Violation of Safety Policy
- ☐ Sample Security Violation
- ☐ Data Entry Error
- ☐ Quality Control Limits Exceeded
- ☐ Check Standards Exceed Limits
- ☐ Data Recording Errors
- ☐ Sample Storage Problem
- ☐ Sample Received at Improper pH
- ☐ Sample Matrix Problem
- ☒ Other (Describe Below)

Anomaly / Incident Description for Laboratory / Field Areas: The high standard (1.0 ppm) displayed an unusually low absorbance value during the analysis of Cold Vapor AA analysis for Mercury. The blank and four other standards appeared to give 'normal' absorbance values. Without using the high (1.0ppm) standard the curve data was acceptable (Correlation coef. = (0.9989), slope = (0.2527), y intercept = (-0.0028). The correlation coefficient using the (1.0ppm) standard was (0.993).

Section 2: Samples / Clients Affected

NLS Sample Numbers: 268700 – 268713 and 269216 – 269225

NLS Project Numbers: 63447 and 63579

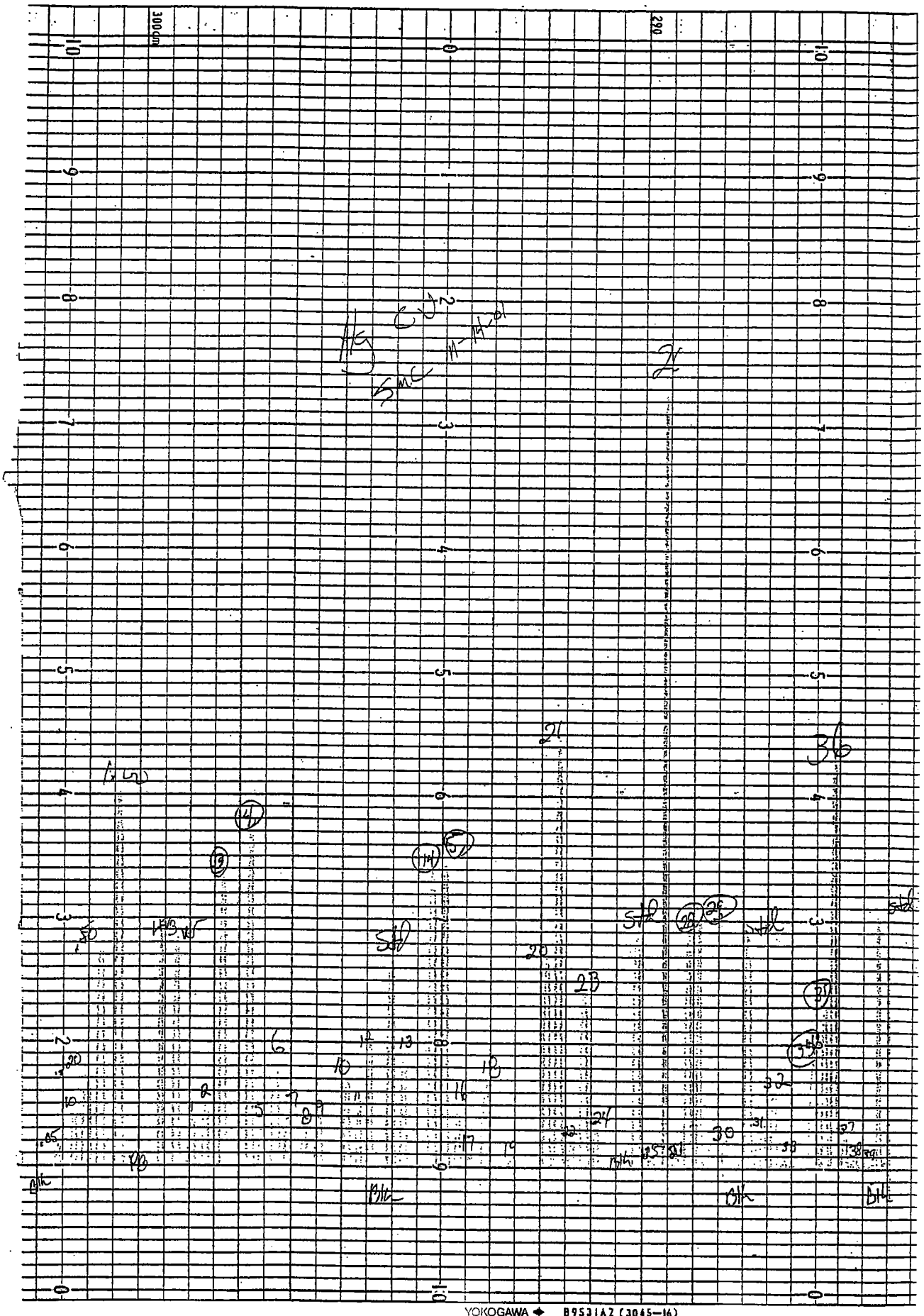
Clients Affected: URS Corporation (Milwaukee)

Section 3: Actions Taken: The (1.0ppm) standard was not used to determine the analytical curve. The curve used had a high standard of (0.5ppm). All analyses that exceeded (0.50ppm) or (0.129) absorbance units were not used and were reanalyzed again on another date.

Information To Be Noted In Client Final Report? (Yes) (No)

Copies To: Area Supervisor, Laboratory Director, QA Officer, Client Files

(Signature) 11/15/2001 (Date)



500143

NORTHERN LAKE SERVICE, INC.

ATTACHMENT 9

LEVEL 4 - QUALITY CONTROL DATA PACKAGE EXAMPLES

AF Cold Vapor - METHOD 245.7M – Mercury (Water)

- NLS AF Cold Vapor QC Data Forms for Mercury Analysis of Water
- NLS Analytical Bench Sheets for AF Cold Vapor Mercury Analysis
- NLS AF Cold Vapor Mercury - Instrument Calibration / QC / Analysis Data Sheets

FORM I INORGANIC ANALYSIS DATA SHEET

Lab Name: Northern Lake Service, Inc

SDG No.:(project) 63781

Lab Sample I.D.: See final report

Matrix (water):

Date Received: See final report

% Solids Not applicable

Date analyzed: 11-29-2001

FORM 2A INITIAL AND CONTINUING CALIBRATION VERIFICATION

Initial Calibration Source: Spex 8-32HG-X

Continuing Calibration Source: Spex 8-32HG-X

Concentration Units: ug/L

Analyte	Initial Calibration			Continuing Calibration					
	True	Found	%R(1)	True	Found	%R(1)	Found	%R(1)	M
Mercury	2.5 ug/L	2.4117	96.5	2.5 ug/L	1 2.4463	97.9	2		245.7M
					3		4		245.7M

FORM 2B LOD STANDARD FOR AA AND ICP not applicable

FORM 3 BLANKS

Preparation Blank Matrix (water):

Preparation Blank Concentration Units (ug/L)

Analyte	Initial Calib. Blank (ug/L)	Q	Continuing Calibration Blank (ug/L)						Preparation Blank		
			CCB #	Q	CCB #	Q	CCB #	Q	Batch #	R	Q
Mercury	ND		1 ND		2		3				
Mercury			4		5		6				

FORM 4 ICP INTERFERENCE CHECK SAMPLE not applicable

FORM 5A SPIKE DUPLICATE

Matrix (water): Level: 1.0 ug/L

Concentration Units (ug/L)

Analyte	Sample Number	Spike Sample Result (SSR)	Q	Sample Result (SR)	Q	Spike Added (SA)	%R	Q	Method
Mercury dig	270703	0.9907		ND		1.0	99.1		245.7M
Mercury dig						1.0			245.7M
Mercury dig						1.0			245.7M
Mercury dig						1.0			245.7M

FORM 7 LABORATORY CONTROL SAMPLE (LFB)

Aqueous LCS Source: Spex 8-17HG

Analyte	Aqueous (ug/L)			
	True	Found	%R	Batch #
Mercury	1.0	1.0653	106.5	
Mercury	1.0			

FORM 8 ICP SERIAL DILUTIONS not applicable

FORM 10 METHOD DETECTION LIMITS

INSTR ID Number: Lachat

Date: 1/20/00

Analyte	Wave length (nm)	Back-ground	LOD (ug/L)	LOQ (ug/L)	Method
Mercury	AF	NA	0.050	0.050	245.7M

FORM 11A ICP INTERELEMENT CORRECTION FACTORS (ANNUALLY) not applicable

FORM 11B ICP INTERELEMENT CORRECTION FACTORS (ANNUALLY) not applicable

FORM 12 ICP LINEAR RANGES not applicable

Type: Metal (AF) - S2 - 103 Holdtime: 21 days Links: PAL .2 : ES 2 : MCL

Notes

Analyzed: 11-29-2001

Analyst M

Checked

Entered

#	INSTRUMENT	LOD	LOQ		DLOD	DLOQ		EPA	SW	SM
1	Hg,dis.af	.05 08/27/96	.05	ug/L	.05 08/27/96	.05	ug/L	245.7M/ 1631M	245.7M/ 1631M	245.7M/ 1631M

MX	TYPE	UNITS	LOVAL	HIVAL	UCL	UWL	MEAN	LWL	LCL	DATE
C	DUP	ug/l	0	3	15.57	11.51	3.41	-4.70	-8.75	08/11/01
C	DUP	ug/l	3	10	8.23	6.21	2.18	-1.85	-3.87	08/11/01
C	DUP	ug/l	10	100	20.00				0.00	
C	SPK	%	0	200	114.82	107.78	93.71	79.64	72.60	08/11/01
LCS	DUP	ug/l	0	3	20.00				0.00	
LCS	DUP	ug/l	3	10	20.00				0.00	
LCS	DUP	ug/l	10	100	20.00				0.00	
LCS	SPK	%	0	200	120.00				80.00	
SC	DUP	ug/l	0	3	20.00				0.00	
SC	DUP	ug/l	3	10	20.00				0.00	
SC	DUP	ug/l	10	100	20.00				0.00	
SC	SPK	%	0	200	120.00				80.00	
W	DUP	ug/l	0	3	21.18	15.81	5.06	-5.68	-11.06	08/11/01
W	DUP	ug/l	3	10	20.00				0.00	
W	DUP	ug/l	10	100	20.00				0.00	
W	SPK	%	0	200	125.60	116.53	98.40	80.26	71.19	03/03/99

BLANK (BLK) - CHECKSTANDARD (CHK) - DUPLICATE (DUP) - RECOVERY (SPK) DATA

SAMPLE#	MTX	TYPE	VALUE1	VALUE2	UNITS	WHO	DATE
270203	C	Spk	99.1		%	M	11-29-2001
270283	C	Dup	0.9907	1.0030	ug/L	M	"

500076

Date & Time Analyzed | 11.29.2001 | Default Units ug/L

[redacted] -- [redacted]
[redacted] - [redacted] - [redacted] - [redacted] - 12.1d-old 9d-hse 09:15

[redacted] -- [redacted]
[redacted] - [redacted] - [redacted] - [redacted] - 12.1d-old 9d-hse 10:00

[redacted] -- [redacted]
[redacted] - [redacted] - [redacted] - [redacted] - 12.1d-old 9d-hse 10:45

[redacted] -- [redacted]
[redacted] - [redacted] - [redacted] - [redacted] - 12d-old 9d-hse 11:45

[redacted] -- [redacted]
[redacted] - [redacted] - [redacted] - [redacted] - 12d-old 9d-hse 12:45

[redacted] -- [redacted]
[redacted] - [redacted] - [redacted] - [redacted] - 12d-old 9d-hse 13:40

[redacted] -- [redacted]
[redacted] - [redacted] - [redacted] - [redacted] - 11.9d-old 9d-hse 14:30

[redacted] -- [redacted]
[redacted] - [redacted] - [redacted] - [redacted] - 11.9d-old 9d-hse 14:55

[redacted] -- [redacted]
[redacted] - [redacted] - [redacted] - [redacted] - 11.9d-old 9d-hse 16:00

[redacted] -- [redacted]
[redacted] - [redacted] - [redacted] - [redacted] - 11.9d-old 9d-hse 16:20

[redacted] -- [redacted]
[redacted] - [redacted] - [redacted] - [redacted] - 11.8d-old 9d-hse 16:45

[redacted] -- [redacted]
[redacted] - [redacted] - [redacted] - [redacted] - 11.8d-old 9d-hse 17:00

[redacted] -- [redacted]
[redacted] - [redacted] - [redacted] - [redacted] - 11.8d-old 9d-hse 17:40

[redacted] -- [redacted]
[redacted] - [redacted] - [redacted] - [redacted] - 11.8d-old 9d-hse 18:10

270201 -- 63781 4 Q Level 4 QC <0.05
[redacted] - GW497025, 30713-20, 30737 - MAX:12/06/01 - MTX:GW - 12.8d-old 8d-hse 16:45

270202 -- 63781 5 Q Level 4 QC <0.05
[redacted] - GW497030, 30721-28, 30738 - MAX:12/06/01 - MTX:GW - 12.7d-old 8d-hse 20:35

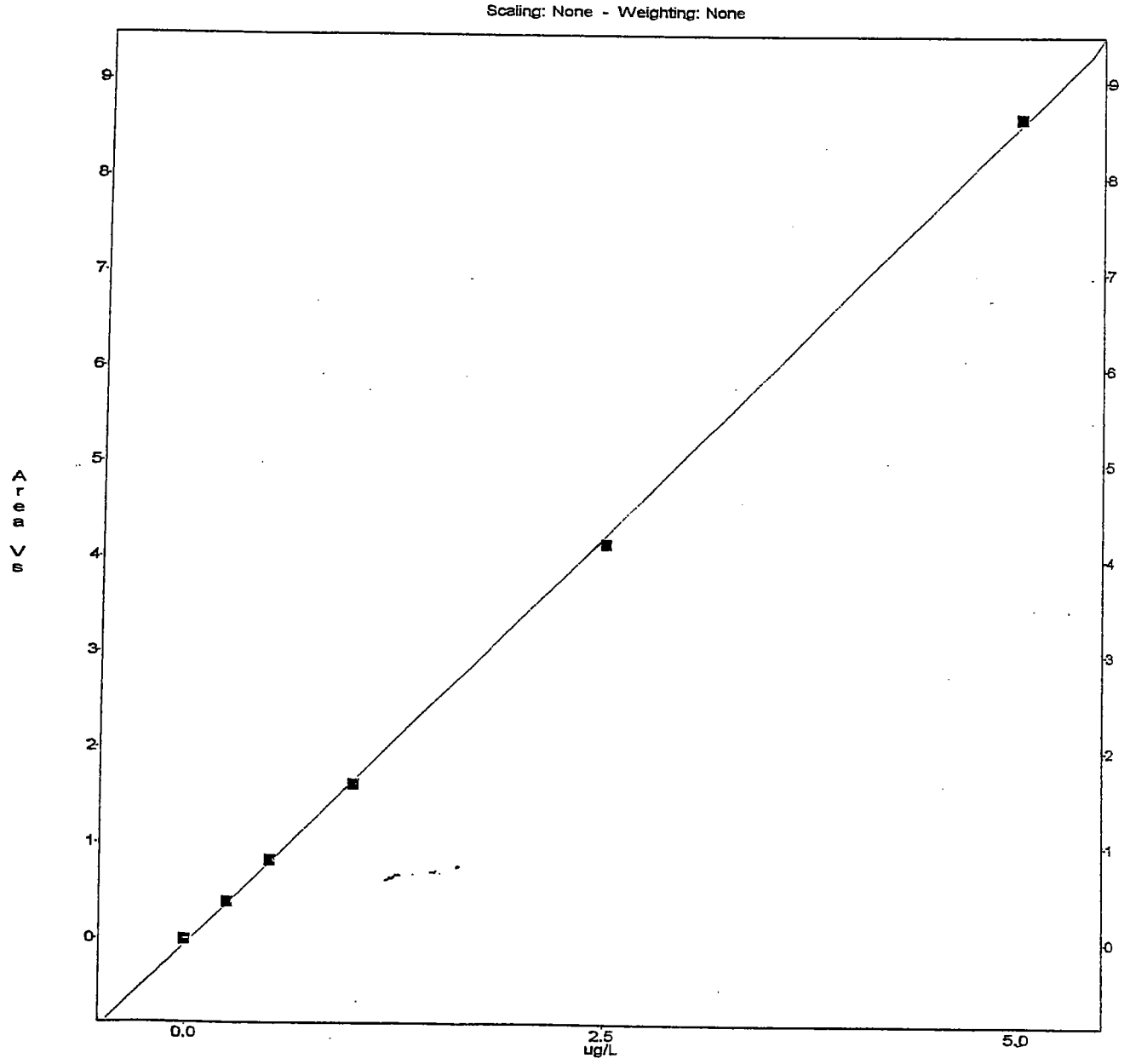
0.9907 1.0030
270203 -- 63781 1 2 Q Level 4 QC 3 <0.05 µg/L 99%
[redacted] - GW497035, 30729-36 - MAX:12/07/01 - MTX:GW - 12d-old 8d-hse 11:50

LFB 6 1.0653 µg/L 106.5%
LRB 7 <0.05 µg/L

Analyte 1

Lvl	Area	ug/L	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Replic STD	Replic % RSD	Residual 1st Poly
1	2616	0.00	2616					0.0	0.0	
2	392489	0.25	392489					0.0	0.0	-3.6
3	829762	0.50	829762					0.0	0.0	-2.5
4	1624661	1.00	1624661					0.0	0.0	2.6
5	4152326	2.50	4152326					0.0	0.0	2.4
6	8621621	5.00	8621621					0.0	0.0	-0.6

1st Order Poly
Conc = 5.801e-007 Area + 3.123e-002
r = 0.9998



Printed: Thursday, November 29, 2001 - 09:33 AM

500078

INSTRUMENT: mercury
TRAY: 011129.TRA METHOD: HGHIGH.MET DATAFILE: 011129.FDT
DATE/TIME: Thu Nov 29 08:49:17 2001 OPERATOR: demo

*** Begin Calibration ***
Cup# 1 Sample: Blank Type: CalStd Level: 1 Rep# 1/1
Ch 1: Analyte 1 Peak Area = 2616.0 µv-s
Cup# 2 Sample: .25 ug/L Type: CalStd Level: 2 Rep# 1/1
Ch 1: Analyte 1 Peak Area = 392489.0 µv-s
Cup# 3 Sample: .5 Type: CalStd Level: 3 Rep# 1/1
Ch 1: Analyte 1 Peak Area = 829762.0 µv-s
Cup# 4 Sample: 1 Type: CalStd Level: 4 Rep# 1/1
Ch 1: Analyte 1 Peak Area = 1624661.0 µv-s
Cup# 5 Sample: 2.5 Type: CalStd Level: 5 Rep# 1/1
Ch 1: Analyte 1 Peak Area = 4152326.0 µv-s
Cup# 6 Sample: 5 Type: CalStd Level: 6 Rep# 1/1
Ch 1: Analyte 1 Peak Area = 8621621.0 µv-s
*** Updated Calibration ***
Ch 1: Analyte 1
** 1st Order Poly Calibration **
C[0] = 5.80063e-007
C[1] = 0.0312333
r = 0.9998
*** End Calibration Block ***
*** Calibration Passed ***
***** Auto DQM Begin *****
*** Starting DQM Set Hghi ***
Cup# 15 Sample: Verification Type: RelChkStd Rep# 1/1
Ch 1: Analyte 1 = 2.4117 ug/L
DQM Sample Results: Verification
Ch 1: Analyte 1 = 2.4117 ug/L
Known Conc = 2.5000 ug/L - %Diff from Known = -3.5318%
Test 1: Passed
Cup# 16 Sample: blank Type: Blank Rep# 1/1
Ch 1: Analyte 1 = 0.0315 ug/L
DQM Sample Results: blank
Ch 1: Analyte 1
Determined Conc = 0.0315 ug/L
Test 1: Passed
*** End of DQM Set Hghi - Set Passed ***
***** Auto DQM End *****
Cup# 1 Sample: 270203 Type: Unknown Rep# 1/1
Ch 1: Analyte 1 = 0.0354 ug/L
Cup# 2 Sample: 270203 spk Type: Unknown Rep# 1/1
Ch 1: Analyte 1 = 0.9907 ug/L
Cup# 3 Sample: 270203 dup spk Type: Unknown Rep# 1/1
Ch 1: Analyte 1 = 1.0030 ug/L
Cup# 4 Sample: 270201 Type: Unknown Rep# 1/1
Ch 1: Analyte 1 = 0.0344 ug/L
Cup# 5 Sample: 270202 Type: Unknown Rep# 1/1
Ch 1: Analyte 1 = 0.0339 ug/L
Cup# 6 Sample: lfb Type: Unknown Rep# 1/1
Ch 1: Analyte 1 = 1.0653 ug/L
Cup# 7 Sample: lrb Type: Unknown Rep# 1/1
Ch 1: Analyte 1 = 0.0314 ug/L
***** Auto DQM Begin *****
*** Starting DQM Set Hghi ***
Cup# 15 Sample: Verification Type: RelChkStd Rep# 1/1 Repeat# 1
Ch 1: Analyte 1 = 2.4463 ug/L
DQM Sample Results: Verification
Ch 1: Analyte 1 = 2.4463 ug/L
Known Conc = 2.5000 ug/L - %Diff from Known = -2.1488%
Test 1: Passed
Cup# 16 Sample: blank Type: Blank Rep# 1/1 Repeat# 1
Ch 1: Analyte 1 = 0.0395 ug/L

DQM Sample Results: blank
Ch 1: Analyte 1
Determined Conc = 0.0395 ug/L
Test 1: Passed
*** End of DQM Set Hghi - Set Passed ***
***** Auto DQM End *****
******* Tray Run Complete *******

NORTHERN LAKE SERVICE, INC.

ATTACHMENT 10

LABORATORY – SPECIFIC STANDARD OPERATING PROCEDURES

- Method 8260 Volatile Organic Compounds (SOP)
- Method 8270 Semi-Volatile Organic Compounds (SOP)
- Method 335.4 Total Cyanide (SOP)
- Method 7196 Hexavalent Chromium (SOP)
- Method 200.7/6010 Trace ICP Metals (SOP)
- Method 200.7/6010 ICP Metals (SOP)
- Determination of Metals by AA – Flame or Furnace (SOP)
- Determination of Mercury by AA Cold Vapor (SOP)
- Determination of Mercury by AF Cold Vapor (SOP)

NORTHERN LAKE SERVICE, INC.

TITLE:

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS BY GC/MS BY EPA METHOD 8260

SOP NUMBER: ORG-METH –GC/MS-8260-1

EFFECTIVE DATE: JAN 3 0 2002

CONTROLLED COPY NUMBER: _____

Originated By: James Deske — NLS Associate — Date: 1-9-02

Approved By: Russ Wolff — Supervisor — Date: 1-9-02

Reviewed By: [signature] — QA Officer — Date: 1/22/2002

Authorized By: [signature] — Laboratory Manager — Date: 1/29/02

I. METHOD TITLE: Determination of Volatile Organic Compounds by GC/MS by EPA Method 8260.

II. METHOD SCOPE AND APPLICATION

A. Parameters

This procedure is capable of determining various volatile organic compounds in a variety of matrices including: groundwater, soil, aqueous sludges, waste solvents, oily wastes, fibrous wastes, soils and sediments. A listing of compounds that may be determined by this procedure along with their MDL values is shown in the appendix of this procedure.

B. NLS Test Codes/Descriptions

C. Detection Limits

1. Aqueous
2. Solid

III. REFERENCES

A. USEPA - Method 8260 B, Revision 2, December 1996. Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition.

IV. METHOD SUMMARY

A. Method 8260 will be used (in conjunction with Method 5030) to quantitate most volatile organic compounds which are insoluble to moderately soluble in water. Volatile compounds are extracted from the sample matrix by bubbling Helium through a 5 ml aqueous sample. The purged components are concentrated on a sorbant trap. When purging has been completed, the sorbant trap is heated and back flushed with Helium onto the GC column. The column is temperature programmed to optimize compound separation. As the compounds elute from the column, they are analyzed by the mass spectrometer (MS).

In some cases, the volatile compounds may be extracted from the sample matrix by solvent with a small portion of the extract being dispersed in water and analyzed.

Qualitative identifications are made based on compound retention times and mass spectra. Quantitative analysis is made by comparison of MS responses to specific ions produced for each compound with another ion produced by an internal standard.

V. INTERFERENCES

A. Matrix/Chemical Interferences

Interferences may be purged or co extracted from the samples and will vary with sample matrix and source. Impurities in the purge gas or sorbant trap and volatile organic compounds in the laboratory are possible contaminant sources. System components which could out-gas into the purge flow stream should be avoided. System contamination should be checked for by daily analysis of a reagent blank. If the blanks are contaminated, the source of contamination should be isolated and eliminated if possible. Subtracting blank values from sample results is not permissible.

Suspected false positives due to laboratory contamination should be explained in the final report sample or case narratives. Samples should be screened if possible.

If a sample with high concentrations results in carry-over contamination, reagent blanks should be analyzed until the system is demonstrated to be clean before sample analysis may resume. The

purge apparatus and sample syringe should be rinsed with two portions of reagent blank grade water between analysis to prevent carry-over of contaminants.

Samples may be contaminated by diffusion of volatile organics through the sample container's septum seal during sample shipment and storage. This contamination source can be checked for by preparation, shipment and analysis of a trip blank.

The methanol content of all standards and samples should be minimized due to methanol quenching of some gas compounds and all late eluters.

VI. SAMPLING

A. Bottle Preparation

1. Samples should be collected in duplicate or triplicate with sample containers filled completely.

B. Preservation

1. When collected, water samples should be acid preserved to a pH of < 2.0 with 1:1 HCl acid.

C. Storage

1. Refrigeration: The VOC sample container should be sealed with the PTFE (side of the) septa down. Refrigerator temperature must be maintained between 1 and 4°C.

D. Holding Times

1. Extraction/Digestion

2. Analysis

a. The samples should come to ambient temperature for analysis.
b. The samples must be analyzed within 14 days of collection.
c. Non-preserved water samples must be analyzed within 7 days.

VII. SAFETY

A. Some of the target compounds are known carcinogens and many others are suspected carcinogens. As a result, samples and standards should be treated as a potential health hazard and exposure to them should be minimized. See the material safety data sheets for additional information.

1. Laboratory arrangement
2. Chemicals
3. Personal

VIII. EQUIPMENT AND MATERIALS

A. Digestion/Extraction/Preparation Equipment

1. Purge & Trap Device: Tekmar LSC 2000 or LSC 3000 and ALS 2016 auto sampler fitted with 5.0 ml fritted sparge tubes.
2. Trap: Supelco 3000 or equivalent.
3. Ultrasonic Cleaner: To be used for soil extraction/sonication.
4. GC: Varian 3400, open/split interface, 1077 Injector, capillary column 75 meter DB 624 or equivalent, and cryogenic cooling controls for liquid CO2 cooling. (Column can be directly interfaced to Tekmar.) To include 60m x .32 mm ID DB-624 1.8 um film thickness or equivalent.
5. Mass Spectrometer: Varian Saturn II, Saturn III or Saturn 2000 & 2000R.

6. Data System: Compaq 486 or better loaded with Saturn software and NIST 92 library, HP Laser Jet Printer.
7. Syringes: 5.0 mL, 1.0 mL, 100 µL, 25 µL, 10 µL, and 5 µL. Syringe valve - two way with luer ends to facilitate sample loading.
8. Balance: Analytical to 0.0001 grams, to include top load capable of weighing 0.01 grams.
9. Volumetric Flasks: Class A - 5 mL, 10 mL, 25 mL, 100 mL, and 1.0 L.
10. Vials: 2 ml and 5 ml with Teflon Faced Septum.
11. Spatula: stainless steel (for soil samples only).

B. Glassware

1. Specifications: 40 ml VOC vial with precleaned silicone PTFE faced septa, Baxter Scientific or equivalent.
2. Preparation

IX. REAGENTS AND STANDARDS

A. Reagent Purity Specifications

1. 1:1: v/v HCL.
2. Methanol: Purge and Trap grade.
3. Water: Organic free, stored in 1.0 L volumetric flasks.
4. Helium: Ultra high purity.
5. Liquid CO2: Sufficient volume to allow a 16 position auto sampler analysis.
6. Internal Standard: Should include pentafluorobenzene, 1,4-difluorobenzene, d5-chlorobenzene, and d4-1,4-dichlorobenzene. Stock solution is normally purchased at 2000 ppm (µg/mL) and diluted to 25 ppm (µg/mL) for a working standard.
7. Surrogate Standards: Should include dibromofluoromethane, d-8 Toluene and 4-bromofluorobenzene. Stock solution is normally purchased at 2000 ppm (µg/mL) and is diluted to 25 ppm (µg/mL) for working standards. A 200 ppm (µg/mL) spiking standard is also maintained for calibration standard preparation.
8. Target Compound Standards: Target compounds are typically purchased at 200 ppm or greater and diluted with methanol as needed to provide working standards of 200 ppm to 1000 ppm.

B. Standards Preparation Directions

Care should be taken to maintain the integrity of all standards. The standards should be kept in sealed ampules or small vials; <5.0 mL with Teflon lined screw caps at temperatures of -10°C to -20°C with minimal exposure to warmer temperatures or light. New standards should be prepared as degradation or expiration dates dictates. Standards may be prepared from neat material.

X. PROCEDURE

A. Extraction/Digestion/Preparation

The ALS should be cleaned by purging each ALS position for at least 5 minutes with elevated line temperatures. Condition the trap by toggling the LSC to bake and hold. The trap should bake for about 15 to 30 minutes prior to daily use and for several hours to condition new traps. Make sure the column is conditioned prior to use and when not in use the GC oven temperature should be maintained at about 60°C with normal helium flow. Ensure the availability of reagent water, standards, and clean purge vessels.

Locate the samples to be analyzed, and read all associated paperwork on them. Also if available, consult back-data on the sample site.

Check the condition of the MS system by checking for air/water in the system, check to ensure available data storage space, purge contaminants from the rough pump oil if necessary, tune and calibrate the MS, and if necessary perform other needed maintenance.

XI. INSTRUMENT ANALYSIS

A. Instrument Settings

1. Tekmar with Supelco 3000 Trap:

Purge flow = 35 cc/min ± 5	Purge Time: 11.0 min
Purge Temp. ambient,	Desorb preheat 250°C
Desorb Temp: 250°C	Desorb Time: 2.0 min.
Bake ≥14 min at 260°C	

2. MS Conditions for Target Acquisition:

Mass range: 35-300 amu	Seconds/scan:0.700 (3 u scans
Acquire time: 38 min	Fil./mult. delay: 160 sec.
Peak Threshold: 0 counts to 2 counts	Mass Defect: -50 mmu/100 amu
Background Mass: 30 amu	Ionization mode: EIA
GC: on	Cal Gas: off
Ion Trap Temperature = 170°C to 220°C	

3. DAILY GC/MS PERFORMANCE TEST:

At the start of each day analysis are to be performed, the GC/MS system must meet the BFB performance criteria. This performance test must be passed prior to any other analysis. BFB (25 – 50 ng) must be injected onto the column and analyzed. The results of the analysis may be corrected for background but must meet the following criteria:

Mass	Ion Abundance Criteria
50	15-40% of mass 95
75	30-60% of mass 95
95	Mass Peak, 100% abundance
96	5-9% of mass 95
173	< 2% of mass 174
174	> 50% of mass 95
175	5-9% of mass 174
176	>95% but < 101% of mass 174
177	5-9% of mass 176

All sample analysis must occur within 12 hours of the BFB analysis.

B. Initial Calibration

The recommended standard concentrations in the curve are 1 ppb, 2.5 ppb, 5.0 ppb, 10.0 ppb, 15.0 ppb and 20.0 ppb. The lowest standard concentration approaching but greater than the method detection limit. The standards should be made in 100 ml volumetrics filled to volume with reagent grade water. After the standards have been spiked into the 100 ml volumetric flasks, cap and invert the flasks three times to mix the contents. The amount of time the flasks are open to atmosphere should always be minimized. When loading the standards onto the Tekmar, always decant the liquid in the neck of the flasks. When transferring the standard into the 5 ml gas tight syringe, minimize the amount of time the standard is exposed to atmosphere and quickly inject the internal standard just before loading.

When the standards have all been analyzed, integrate the peaks and calibrate to each standard level. Observe the response factors (RFs) for each compound and check any figures which seem to be unusually high or low. The elution of methanol may affect the responses of bromomethane and chloroethane adversely. The spectrum of chloroethane will always be distorted due to the co-elution of water and methanol. The average RF of the SPCC and CCC compounds must also be carefully checked. The 5 system performance check compounds (SPCCs) are chloromethane, 1,1-dichloroethane, bromoform $\geq$ 0.100, chlorobenzene and 1,1,2,2-Tetrachloroethane $\geq$ 0.300. Failure of these criteria requires system repairs and recalibrations.

1. Chloromethane RF is low - This compound is most likely to be lost if purge flow is too high. It may also be lost in system leaks/contamination or demonstrate the need for a new sorbant trap.
2. Bromoform RF is low - This compound responds poorly with low purge flows. It also can indicate cold spots or active sites on transfer lines, and may indicate the need for a new sorbant trap.
3. 1,1,2,2-Tetrachloroethane and 1,1-dichloroethane are prone to degradation by contaminated transfer lines in the purge and trap system or on active sites in trapping materials.

C. Every reasonable effort should be made to keep the % RSDs for all compounds less than 15% to promote the acquisition of good sample data and maintain QC limits with relatively low tolerances for error. Additionally % RSDs must be $\leq$ 15.0 to use average RF for calculation of concentrations. If % RSDs are greater than 15% a higher order regression fit may be used for calculations or a new curve must be run. Also the % RSDs for CCCs must be less than 20 in order to consider a curve valid.

D. DAILY GC/MS CALIBRATION: (Calibration Check Standard)

Calibration check compounds must be < 20% difference from the initial calibration responses.

1. $$\% D = \frac{(RFi - RF)c}{(RFi)} X\ 100$$

The CCCs are Vinyl chloride, 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, and Ethylbenzene.

Internal standard retention times and areas should be evaluated in the check standard. The retention time for internal standards must not vary by more than 30 seconds from the previous calibration check standard. Internal standard peak areas should be 50 to 200% of those in the initial calibration. Every reasonable effort should be made to keep the % Differences for all compounds less than 20% to promote the acquisition of good sample data and maintain good quality control. All target compounds should be $\leq$ 20 % difference from the initial calibration RFs.

2. METHOD BLANK

A blank made up with reagent water and spiked with internal standard and surrogate standards to 10 ppb should be analyzed prior to sample analysis but after the standards. The method blank is analyzed to check for system, water, or standard contamination. It is possible to find Methylene Chloride, Acetone, 2-Butanone or Toluene in the blank, particularly if these common laboratory solvents are in use somewhere in the laboratory. Blank values for any compounds found in the blank should be noted if at or above the detection limits. Blank values are not to be subtracted from sample values. Every effort should be make to minimize contamination in the laboratory.

3. WATER SAMPLE ANALYSIS

If possible, samples can be screened prior to analysis. Samples should be analyzed under the same conditions as the standard and blank. Back data on project sites should be used to establish approximate dilutions necessary.

Prior to analysis and loading all samples should be allowed to come to ambient temperature. Any sample which has been opened or has headspace should not be analyzed due to the loss of sample integrity. If analyzed, the sampled must be qualified on the final report.

The GC/MS must pass the BFB criteria and the initial and/or continuing calibration prior to the analysis of samples. Also, a method blank must be analyzed prior to any samples. Any dilutions or composites may be accomplished within a volumetric flask or within a 5.0 mL air-tight syringe. The time that the sample vials, volumetrics and syringes are left open to the atmosphere should be minimized at all times. All sample dilutions should be in the upper one-half of the calibration range. If an analysis results in saturated ions, a blank or series of blanks must be run until the system is free of interference.

When the 5.0 mL gas-tight syringe has been filled with sample and adjusted to 5.0 mL, the sample should be spiked with 10 ppb internal and surrogate standards, (2.0 µL of each at 25 ppm). Matrix spikes should be analyzed at the 10 ppb concentration level. The matrix spike mix should include all target analytes in order to maximize the quality control information on the samples. A matrix spike/spike duplicate should be performed on each analysis day.

Water samples should be purged, desorbed and analyzed in exactly the same manner as all standards and blanks associated with the water samples. Methanol has a quenching effect on many compounds and should not be added in amounts over 50 µLs to 5.0 mL of water.

4. **SOIL, SEDIMENT AND WASTE SAMPLES**

All soil samples are methanol preserved in accordance with WDNR instructions. (The only exception to the methanol preservation rule will be solids that will be extracted by the laboratory.) Soil samples should be collected and stored in tare weighted VOC soil jars and field preserved with methanol in an approximate 1 mL to 1 g of soil ratio. The laboratory will then adjust the ratio of slightly underpreserved samples to 1:1 before further preparations for analysis. Note that some fibrous wastes and sludges do not lend themselves to this type of preservation and will need to be methanol extracted by the laboratory.

Soil Extraction Method: NLS will supply 60 mL wide mouth vials with Teflon lined caps in duplicate. Also supplied will be 25 mL vials of methanol. As close to 25 grams as possible of soil is to be added to the jar. The 25mL vial of methanol is to be opened and added immediately to preserve and extract the VOC's from the soil. Samples are to be stored at 4 degrees C. The sample must be analyzed within 21 days and any confirmations must be done within 28 days. Up to 50 uLs of methanol can be analyzed in 4.95 mL's of water.

F. Sample/Standard Presentation to Instrument – See Section XI E. 2.

XII. CALCULATIONS

A. DATA INTERPRETATION:

When data acquisition has been completed the sample data may be integrated. The samples must be integrated against the initial calibration data to ensure that the proper retention times, RFs and spectra are used. Ion relative intensities must match to within ± 20% of the standard spectra used in calibrations and all ions above 10% in the standard spectra must be present in the sample spectra for a positive identification.

Also, methanol based samples must be integrated against the methanol based initial calibrations and waters must be integrated against the water initial calibration. When integration has been completed the results may be quantitated by comparing target ion abundances (areas), against internal standard ion areas and initial calibration response factors as follows:

$$\text{Concentration} = \frac{\text{Area Target Ion}}{\text{Area of Internal Standard}} \text{ X } \frac{\text{Amount of Internal Standard Added}}{\text{Average RF from Initial Calibration}}$$

These results are corrected for dilutions, extractions and percent solids when data is entered into the LIMS system. On occasion the computer may "miss" compounds during the spectrum search phase of integration for any number of reasons. In such instances, the target compound quantitation ion is integrated as well as the internal standard ion, and hard copies are made. The concentration formula is then used to determine the concentration of the target compound.

If there is an obvious interference with the quantitation ion(s). A secondary ion is chosen for integration and the response factor is calculated from the initial calibration referenced on that analysis day. Unknown compounds found during the verification of spectra following quantitation are integrated along with the nearest internal standard free of interferences. The concentration of this unknown is established by the following formula:

$$\text{Concentration} = \frac{\text{Total Peak Area of Unknown}}{\text{Total Peak Area of Internal Standard}} \text{ X } \frac{\text{Concentration of Internal Standard}}{\text{Assumed RF of 1.000}}$$

The concentration of the unknown in the sample analysis must be at least 1.0 ppb before corrections for dilutions etc. in order to be reported out as a Tentatively Identified Compound (TIC). Those compounds which make the 1.0 ppb cutoff are then searched against the NIST92 spectral library for identification and the operator then will attempt to establish the best identification by spectrum and retention time. The concentrations of TICs are corrected for dilutions, extractions and percent solids during data entry into the LIMS system. This laboratory will not report TICs unless requested for by our clients.

B. Significant figures – Report to two significant figures.

XIII. QUALITY CONTROL

A. The GC/MS system must meet BFB specifications each 12 hours of operations. The initial calibration must meet all specifications and the calibration standards must meet all specifications for SPCC and CCC criteria each 12 hour day of operations. Each time a new operator is trained to operate GC/MS or major changes are made in instrumentation an initial demonstration of proficiency must be performed. Additionally before any samples are analyzed, a method detection limit study must be performed.

Internal standards recoveries of calibration checks must be 50-200% of those in the initial calibration. Additionally internal responses of samples should not vary by more than 30% from the calibration check standard.

Surrogate standard recoveries should fall within laboratory established QC limits. Samples with internal or surrogate standards outside of the QC limits must be reanalyzed if no interferences or miscalculations are present. If the reanalysis is also outside of the QC limits, then the results should be reported "as is" and flagged.

The laboratory should maintain a QC database for target compound recovery limits in spikes and duplicate spikes. Samples containing analytes which are outside of QC limits in the spikes should be reanalyzed if time permits. If time does not permit reanalysis or the recovery problem persists, the data should be flagged.

The laboratory should perform an MDL study on approximately a yearly basis. The MDL study may be used as an initial demonstration of capability if one of the MDL study levels will meet the criteria.

XIV. ATTACHMENTS

A. Compound/Analyte list and MDLs

B. Worksheets or forms for recording data

CHARACTERISTIC MASSES (M/S) FOR PURGEABLE ORGANIC COMPOUNDS

Analyte	Primary Quantitation Ion	Secondary Quantitation Ion(s)
Benzene	78	--
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon tetrachloride	117	119
Chlorobenzene	112	77, 114
Chloroethane	Sum of 45 + 49	--
Chloroform	83	85
Chloromethane	50	52
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61,98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77
trans-1,3-Dichloropropene	75	77
Ethylbenzene	91	106
Hexachlorobutadiene	225	223, 227
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Methylene chloride	84	86, 49
Naphthalene	128	--
n-Propylbenzene	91	120
Styrene	104	78
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	166	168, 129
Toluene	92	91
1,2,3-Trichlorobenzene	180	182, 145

1,2,4-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	130, 132
Trichlorofluoromethane	101	103
1,2,3-Trichloropropane	110	77, 75
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl chloride	62	64
o-xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
INTERNAL STANDARDS/SURROGATES		
4-Bromofluorobenzene	95	174, 176
Dibromofluoromethane	113	111
Toluene-d8	98	100
Pentafluorobenzene	168	--
1,4-Difluorobenzene	114	--
Chlorobenzene-d5	117	--
1,4-Dichlorobenzene-d4	152	--
MAY ALSO INCLUDE:		
Acetone	43	58
Acrolein	55	56
Acrylonitrile	52	53
2-Butanone	43	72
Carbon Disulfide	76	--
2-Chloroethyl vinyl ether	63	65, 106
2-Hexanone	43	58, 57, 100
Vinyl Acetate	43	--
Methyl-tert-Butyl Ether	73	--
4-Methyl-2-Pentanone	43	58, 57, 100
*Tetrahydrofuran	71	41
Iodomethane	142	127
Trans-1,4-Dichloro-2-butene	88	124
*2,3-Dichloro-1-propene	75	110
Dichlorofluoromethane	67	69
Ethyl Ether	59	45
*112-Trichloro-122-trifluoroethane	151	101
Allyl Chloride	76	--
*Dichloroacetronitrile	74	--
Chloroprene	88	88
Methyacrylonitrile	67	--
Methyl Methacrylate	99	69
Ethyl Methacrylate	99	69

*Not officially on the compound list for 8260B but can be determined and flagged as not within the scope of 8260B.

APPENDIX - A

METHOD 8260/8240 COMPOUND AND CAS NUMBER LISTING

Analyte	CAS Number
Acetone	67-64-1
Acrolein	107-02-8
Acrylonitrile	107-13-1
Allyl Chloride	107-05-1
Benzene	71-43-2
Bromobenzene	108-86-1
Bromochloromethane	74-97-5
Bromodichloromethane	75-27-4
4-Bromofluorobenzene (surrogate)	460-00-4
Bromoform	75-25-2
Bromomethane	74-83-9
2-Butanone	78-93-3
n-Butylbenzene	104-51-8
sec-Butylbenzene	135-98-8
tert-Butylbenzene	98-06-6
Carbon disulfide	75-15-0
Carbon Tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chlorobenzene-d5 (internal std.)	108-90-7
Chlorodibromomethane	124-48-1
Chloroethane	75-00-3
2-Chloroethyl Vinyl Ether	110-75-8
Chloroform	67-66-3
Chloromethane	74-87-3
Chloroprene	126-99-8
2-Chlorotoluene	95-49-8
4-Chlorotoluene	106-43-4
1,2-Dibromo-3-Chloropropane	96-12-8
Dibromofluoromethane (surrogate)	-------
1,2-Dibromomethane	106-93-4
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
Dibromomethane	74-95-3
d4-1,4-Dichlorobenzene (internal std.)	-------
Trans-1,4-Dichloro-2-butene	764-41-0
Dichlorodifluoromethane	75-71-8
Dichlorofluoromethane	
1,1-Dichloroethane	75-34-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethene	75-35-4
cis-1,2-Dichloroethene	156-59-4
trans-1,2-Dichloroethene	156-60-5
1,2-Dichloropropane	78-87-5
1,3-Dichloropropane	142-28-9
2,2-Dichloropropane	594-20-7
1,1-Dichloropropene	563-58-6
cis-1,3-Dichloropropene	10061-01-5

trans-1,3-Dichloropropene	10061-02-6
2,3-Dichloro-1-propene	78-88-6
1,4-Difluorobenzene (internal std.)	540-36-3
Ethylbenzene	100-41-4
Ethyl Ether	60-29-7
Ethyl methacrylate	97-63-2
2-Hexanone	591-78-6
Iodomethane	74-88-4
Isopropylbenzene	98-82-8
p-Isopropyltoluene	99-87-6
Methacrylonitrile	126-98-7
Methylene Chloride	75-09-2
Methyl methacrylate	80-62-6
4-Methyl-2-pentanone	108-10-1
Methyl-tertbutyl ether	1634-04-4
Pentachlorobenzene (internal std.)	--------
Styrene	100-42-5
1,1,1,2-Tetrachloroethane	630-20-6
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethene	127-18-4
Tetrahydrofuran	109-99-9
Toluene	108-88-3
Toluene-d8 (surrogate)	108-88-3
1,2,3-Trichlorobenzene	87-61-6
1,2,4-Trichlorobenzene	120-82-1
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethene	97-01-6
Trichlorofluoromethane	75-69-4
1,2,3-Trichloropropane	96-18-4
1,2,4-Trimethylbenzene	95-36-3
1,3,5-Trimethylbenzene	108-67-8
Vinyl Acetate	108-05-4
Vinyl Chloride	75-01-4
Xylene (total)	1330-20-7

END

NAME	ESORT	PEAK	LOD	LOQ	UNITS	PCOD	RSOR
SAT2WX	4	Dichlorodifluoromethane	.16	.508	ug/L	34668	24
SAT2WX	5	Chloromethane	.238	.757	ug/L	34418	14
SAT2WX	6	Vinyl chloride	.174	.555	ug/L	39175	58
SAT2WX	6.5	1,3-Butadiene	.129	.409	ug/L	q	123
SAT2WX	7	Bromomethane	.21	.667	ug/L	34413	6
SAT2WX	8	Chloroethane	.667	2.123	ug/L	34311	12
SAT2WX	9	Trichlorofluoromethane	.166	.527	ug/L	34488	54
SAT2WX	9.5	Dichlorofluoromethane	.2	.637	ug/L	q	124
SAT2WX	9.6	Ether	.437	1.389	ug/L	q	125
SAT2WX	10	Acrolein	1.271	4.389	ug/L	34210	102
SAT2WX	11	1,1-Dichloroethene	.162	.515	ug/L	34501	27
SAT2WX	11.5	1,1,2-Trichlorotrifluoroethane	.145	.461	ug/L	81611	126
SAT2WX	12	Iodomethane	.122	.387	ug/L	77424	99
SAT2WX	13	Acetone	5.5	5.5	ug/L	81552	98
SAT2WX	14	Carbon disulfide	.212	.674	ug/L	77041	100
SAT2WX	14.5	Isopropyl alcohol	7.628	24.27	ug/L	q	149
SAT2WX	15	Allyl chloride	.186	.592	ug/L	78109	111
SAT2WX	16	Acetonitrile	1.904	6.057	ug/L	76997	116
SAT2WX	17	Methylene chloride	.257	.818	ug/L	34423	40
SAT2WX	18	Acrylonitrile	.625	1.989	ug/L	34215	101
SAT2WX	19	trans-1,2-Dichloroethene	.22	.699	ug/L	34546	29
SAT2WX	20	MTBE	.285	.906	ug/L	78032	97
SAT2WX	21	Hexane	.171	.545	ug/L	81590	117
SAT2WX	22	1,1-Dichloroethane	.231	.736	ug/L	34496	25
SAT2WX	23	Chloroprene	.211	.67	ug/L	81520	112
SAT2WX	24	Vinyl acetate	.857	2.958	ug/L	77057	103
SAT2WX	24.5	Isopropyl ether	.296	.941	ug/L	81577	130
SAT2WX	25	2,2-Dichloropropane	.16	.51	ug/L	77170	32
SAT2WX	26	cis-1,2-Dichloroethene	.243	.772	ug/L	77093	28
SAT2WX	27	Methyl ethyl ketone	.85	2.703	ug/L	81595	104
SAT2WX	28	Propionitrile	2.95	10.185	ug/L	77007	118
SAT2WX	28.5	Ethyl acetate	1.712	5.913	ug/L	q	131
SAT2WX	29	Methacrylonitrile	.499	1.588	ug/L	81593	113
SAT2WX	30	Bromochloromethane	.223	.71	ug/L	77297	3
SAT2WX	31	Tetrahydrofuran	.626	1.99	ug/L	81607	108
SAT2WX	32	Chloroform	.23	.73	ug/L	32106	13
SAT2WX	33	1,1,1-Trichloroethane	.218	.693	ug/L	34506	51
SAT2WX	34	Carbon Tetrachloride	.116	.37	ug/L	32102	10
SAT2WX	35	1,1-Dichloropropene	.105	.349	ug/L	77168	33
SAT2WX	36	Isobutanol	5.665	18.023	ug/L	77033	119
SAT2WX	37	Benzene	.162	.516	ug/L	34030	1
SAT2WX	38	1,2-Dichloroethane	.196	.623	ug/L	32103	26
SAT2WX	39	Heptane	.182	.579	ug/L	q	120
SAT2WX	40	Trichloroethene	.136	.432	ug/L	39180	53
SAT2WX	41	1,2-Dichloropropane	.154	.49	ug/L	34541	30
SAT2WX	42	2,3-Dichloro-1-propene	.188	.597	ug/L	q	109
SAT2WX	43	Methyl methacrylate	.486	1.545	ug/L	81597	114
SAT2WX	44	Dibromomethane	.144	.458	ug/L	77596	20
SAT2WX	45	Bromodichloromethane	.166	.53	ug/L	32101	4
SAT2WX	46	2-Chloroethyl vinyl ether	.181	.576	ug/L	34576	105

NAME	ESORT	PEAK	LOD	LOQ	UNITS	PCOD	RSOR
SAT2WX	47	cis-1,3-Dichloropropene	.149	.474	ug/L	34704	34
SAT2WX	48	4-methyl-2-pentanone	.567	1.805	ug/L	78133	106
SAT2WX	49	Toluene	.129	.411	ug/L	34010	48
SAT2WX	50	Octane	.166	.527	ug/L	q	121
SAT2WX	50.5	Dichloroacetonitrile	2.33	8.046	ug/L	q	127
SAT2WX	51	trans-1,3-Dichloropropene	.147	.469	ug/L	34699	35
SAT2WX	52	Ethyl methacrylate	.228	.725	ug/L	73570	115
SAT2WX	53	1,1,2-Trichloroethane	.192	.609	ug/L	34511	52
SAT2WX	53.5	4,4-Dimethyl-2-pentanone	1.344	4.278	ug/L	q	128
SAT2WX	54	Tetrachloroethene	.125	.399	ug/L	34475	47
SAT2WX	55	1,3-Dichloropropane	.227	.722	ug/L	77173	31
SAT2WX	56	2-Hexanone	.907	2.887	ug/L	77103	107
SAT2WX	57	Dibromochloromethane	.195	.619	ug/L	32105	17
SAT2WX	58	1,2-Dibromoethane	.186	.593	ug/L	77651	19
SAT2WX	59	Chlorobenzene	.163	.519	ug/L	34301	11
SAT2WX	60	1,1,1,2-Tetrachloroethane	.194	.617	ug/L	77562	45
SAT2WX	61	Ethylbenzene	.159	.505	ug/L	78113	36
SAT2WX	62	meta,para-Xylene	.209	.666	ug/L	85795	59
SAT2WX	63	ortho-Xylene	.125	.399	ug/L	77135	43
SAT2WX	64	Styrene	.126	.402	ug/L	77128	44
SAT2WX	65	Bromoform	.358	1.238	ug/L	32104	5
SAT2WX	66	Isopropylbenzene	.128	.408	ug/L	77223	38
SAT2WX	67	cis-1,4-Dichloro-2-butene	.186	.622	ug/L	q	122
SAT2WX	67.5	Cyclohexanone	3.643	11.589	ug/L	q	132
SAT2WX	68	1,1,2,2-Tetrachloroethane	.383	1.322	ug/L	34516	132
SAT2WX	69	Bromobenzene	.204	.649	ug/L	81555	2
SAT2WX	70	1,2,3-Trichloropropane	.313	.997	ug/L	77443	55
SAT2WX	71	Trans 1,4-dichloro 2-butene	.457	1.455	ug/L	73547	110
SAT2WX	72	n-Propylbenzene	.136	.432	ug/L	77224	42
SAT2WX	73	2-Chlorotoluene	.143	.456	ug/L	77275	15
SAT2WX	74	1,3,5-Trimethylbenzene	.139	.441	ug/L	77226	57
SAT2WX	75	4-Chlorotoluene	.104	.332	ug/L	77277	16
SAT2WX	76	tert-Butylbenzene	.106	.338	ug/L	77353	9
SAT2WX	77	1,2,4-Trimethylbenzene	.118	.376	ug/L	77222	56
SAT2WX	78	sec-Butylbenzene	.14	.446	ug/L	77350	8
SAT2WX	79	1,3-Dichlorobenzene	.155	.493	ug/L	34566	22
SAT2WX	80	p-Isopropyltoluene	.211	.727	ug/L	77356	39
SAT2WX	81	1,4-Dichlorobenzene	.13	.415	ug/L	34571	23
SAT2WX	82	n-Butylbenzene	.144	.46	ug/L	77342	7
SAT2WX	83	1,2-Dichlorobenzene	.165	.524	ug/L	34536	21
SAT2WX	84	1,2-Dibromo-3-Chloropropane	.486	1.677	ug/L	38437	18
SAT2WX	85	1,2,4-Trichlorobenzene	.153	.486	ug/L	34551	50
SAT2WX	86	Hexachlorobutadiene	.271	.862	ug/L	34391	37
SAT2WX	87	Naphthalene	.293	.933	ug/L	34696	41
SAT2WX	88	1,2,3-Trichlorobenzene	.168	.533	ug/L	77613	49

NORTHERN LAKE SERVICE
SATURN 2 - GC/MS INSTRUMENT RUN LOG
8260 VOLATILES ANALYSIS LOGBOOK

BOOK:_____
PAGE:_____

Date:	Method:	Analyst:	Initial Calibration Area Counts: Pentafluorobenzene:	1,4-Difluorobenzene:	Chlorobenzene-d5:	1,4-Dichlorobenzene-d4:	Calibration Date:
	8260						

File No.	Sample Number	Stn. No.	Sample Amount	Dil. Fact.	pH< 2 Y/N	Pentafluorobenzene (I.S.) [Area Counts]	1,4-Difluorobenzene (I.S.) [Area Counts]	Chlorobenzene-d5 (I.S.) [Area Counts]	1,4-Dichlorobenzene-d4 (I.S.) [Area Counts]	Dibromo-fluoromethane [S.S. %Rec.]	Toluene-d8 (S.S.) [% Rec.]	BFB (S.S.) [% Rec.]
1	VBFB / /											
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												

Comments / Repeats:	Standards Reference	25 ppm (I.S.)	25 ppm (S.S.)	200 ppm (S.S.)	Acrolein Vinyl Acetate	Custom VOCs	502.2 Mix					
	Book #:											
	Page #:											
	Line #:											
Project Numbers:	Run Time:			To:								

DATE

ANALYST

NORTHERN LAKE SERVICE
ORGANIC EXTRACTION LOG
METHANOL PRESERVATION (SOILS)
VOLATILES ANALYSIS

BOOK:
PAGE:

Project #	Sample Number	Tare Weight (grams)	Final Weight (grams)	MeOH Weight (grams)	**Soil Weight (grams)**	MeOH Added (mLs)	Comments

SAMPLES SONICATED FOR 20 MINUTES: YES ***Analyst:*** _____

Comments:

_____

_____

_____

NORTHERN LAKE SERVICE, INC.

TITLE:

DETERMINATION OF SEMI-VOLATILES USING GC/MS EPA METHOD 8270C

SOP NUMBER: ORG-METH-GC/MS-S/VOL-1

EFFECTIVE DATE: JAN 1 7 2002

CONTROLLED COPY NUMBER: _____

Originated By: Ron Wolff — NLS Associate — Date: 11-05-01

Approved By: Jerry R Booth — Supervisor — Date: 01/07/02

Reviewed By: [signature] — QA Officer — Date: 1/8/2002

Authorized By: [signature] — Laboratory Manager — Date: 1/16/02

I. METHOD TITLE: Determination of Semi-Volatiles using GC/MS EPA Method 8270C

II. METHOD SCOPE AND APPLICATION

The following SOP is designed to meet the analytical requirements of SW-846 Method 8270C.

Method 8270C is used to determine the concentration of Semi-Volatile organic compounds in extracts prepared from all types of solid waste matrices, soils, and ground water.

Method 8270C can be used to quantitate most neutral, acidic and basic compounds that are soluble in methylene chloride and capable without derivatization of producing sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphorus esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.

The information contained in this Standard Operating Procedure is in reference to the GC-MSD system by Hewlett-Packard, model 5972A. The GC is a Hewlett-Packard model 5890, series II with electronic pressure control.

A. Parameters: See attachment I for a complete listing of the compounds which may be determined by Method 8270C. The WDNR has given NLS permission to run Semi-volatile compounds not included in the published list as long as MDLs have been determined.

B. NLS Test Codes/Descriptions

1. testcode 15094/8270C Water Analysis
2. testcode 15084/8270C Soil Analysis
3. testcode 15091/3510C (BNA) Extraction
4. testcode 15092/3510B (Acid) Extraction
5. testcode 15093/3510C (BN) Extraction
6. testcode 11270/3520C Continuos Liq-Liq Extraction

C. Detection Limits

1. See attachment II for a complete listing of the current MDLs for aqueous and solid samples.

III. REFERENCES

A. Method 8270C – Revision 3 – Semi-volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS).

B. SW-846, Update III, December 1996.

IV. METHOD SUMMARY

A. Gas Chromatography: Chromatography is essentially a physical method of separation in which components to be separated are distributed between two phases, one of them constituting a stationary phase and the other being the mobile phase. Chromatographic separation occurs as a result of repeated sorption-desorption acts during the movement of sample components along the stationary phase.

In Gas Chromatography the sample is volatilized in the injector port and swept onto the stationary phase of the column by the mobile gas phase (Helium). The separation takes place as the analytes

travel along the column and interact with the column stationary phase. Analytes emerge at the end of the column at different times and enter the detector where they produce a signal proportional to the sample concentration.

The purpose of the injector port is to vaporize the liquid samples and permit the carrier gas to push the vapor in the form of a concentrated plug onto the beginning of the column with a minimum of time lapse. The sample introduction system consists of a heated block containing a glass insert which funnels the vapor onto the column. The liquid samples are injected into the glass insert with a 10 microliter syringe which penetrates the closed system by piercing a pliable synthetic septa which reseals the system after the needle is withdrawn.

The column is housed in a temperature programmable oven which is heated at a rate optimized to produce sufficient peak resolution in the shortest time possible. Heating the oven will speed up the compounds interaction with the stationary phase causing it to emerge at the end of the column more quickly than it would at a lower temperature.

The initial oven temperature, the oven temperature ramp rate, final oven temperature and carrier gas Flow rate are all variables which influence the separation process.

B. Mass Spectrometry: The mass spectrometer can be divided into the following main parts:

1. Sample Inlet Interface.
2. Ion Source.
3. Mass Filter.
4. Ion Detector.
5. Data Handling System.
6. Vacuum Chamber and Pumping System.

The Inlet system interface is a heated path from the GC oven to the MSD Ion Source. The interface is mounted on the right side of the vacuum manifold. One end of the interface passes through the side of the gas chromatograph and protrudes into the GC oven. The other end of the interface fits into a socket on the Ion Source side in the analyzer. The interface is heated by an electric cartridge heater and should be operated in the range of 250 to 320 degrees centigrade. The set point should be operated slightly higher than the maximum GC oven temperature to avoid condensation of higher-boiling analytes.

The Ion Source is an Electron Impact (EI) source. It has a cylindrical geometry, which ensures proper concentric alignment of the Ion Source elements. The sample enters the Ion Source from the GC interface. Electrons emitted by one of the filaments enter the Ion Source volume, which is at ground potential. A small permanent magnet focuses the electron beam. The electrons ionize and fragment the sample molecules. Positive and negative ions are generated. Positive ions are repelled from the Ion Source volume and pass through several electromagnetic lenses. These lenses concentrate the ions into a tight beam and direct them into the Mass Filter.

The Mass Filter allows selected ions from the Ion Source to reach the detector. When correctly tuned it filters and separates ions based on their mass to charge ratio. Only an ion of one mass can pass through the filter at a given time. Once the ions are inside the Mass Filter they experience the rapidly oscillating RF voltages. Ions which are not mass selected become synchronized with the oscillating RF field and are catastrophically accelerated into the rods.

The quadrupole Mass Filter may be operated in one of two modes. Scan or SIM (Selected Ion Monitoring). In SIM the Mass Filter is set to pass one kind of ion. This provides the greatest sensitivity and is used in quantitative applications. It is used when the analyst has prior knowledge of what ions to expect. In Scan mode the Mass Filter is set to sequentially operate through a range of masses. It has lower sensitivity because most of the ion strike the rods during the scan.

An Ion Detector is used to collect and count ions or detect their resulting current. Only one mass to charge ion is detected at a given time. When the mass selected ion collides with the detector the resulting signal can be displayed by the data system

The data system sends information to the Mass Spectrometer electronics. This determines in part how the inlet, ion source, mass filter detector and vacuum system operate. The data system also stores and collects the resultant signal from the ion detector. This information can be displayed real time or stored for later data processing.

The Ion Source, Mass Filter and Detector are in vacuum so that a collision of a given ion with another molecule such as air or water is unlikely. Minimizing collisions ensures that the ions are efficiently mass selected and detected. Common vacuum systems consist of two kinds of pumps. A high vacuum pump and a rough pump. Initially the rough pump pulls out most of the air/water and other gaseous species. The high vacuum pump acts to concentrate the rarified high vacuum gasses which are subsequently removed by the rough pump making mass spectral analysis possible.

I. INTERFERENCES

A. Matrix/Chemical Interferences

By nature, GC/MS is designed to limit the misidentification of compounds, however there is a potential for improperly reporting "compounds" that show up in the quantitation report. Therefore, all "compounds" must be verified through a careful inspection of the spectra. Some compounds have almost identical spectra and similar elution times. These compounds can be mislabeled if the analyst is not careful in determining which compound is actually present. Some of these compounds pairs are listed as follows: *2,4-dichlorophenol & 2,6-dichlorophenol, 1-chloronaphthalene & 2-chloronaphthalene, 1-naphthalene & 2-naphthalene, 2,4,6-trichlorophenol & 2,4,5-trichlorophenol, Phenanthrene & Anthracene, Benzo(a)anthracene & chrysene, Benzo(b)fluoranthene & Benzo(k)fluoranthene.* These peak pairs can be distinguished using relative retention times if they appear as a single peak, assuming the elution order of the column has been previously determined by injecting the individual analytes

Raw GC/MS data files from all blanks, samples, and spikes must be evaluated for interference. Determine if the contamination source is in the preparation and/or cleanup of samples and take corrective action to eliminate the problem. Phthalates are common lab contaminants for semi-volatiles. Phthalates and adipates are plasticizers used to soften plastic and are found virtually everywhere. It is advisable to eliminate all sample contact with plastic being careful not to expose the glassware to any source of phthalates, such as soap, etc.

VI. SAMPLING

A. Bottle Preparation: Aqueous samples should be collected in two, 1 Liter glass amber containers with Teflon lined caps. Solid samples should be collected in one 6 oz. glass soil jars with a Teflon lined cap. These quantities are sufficient for replicate analysis. Plastic must be avoided to prevent phthalate contamination.

B. Preservation: Sodium Thiosulfate preservation is required if residual chlorine is present in the sample. Storage at 1 - 4 degrees centigrade is necessary for sample transport and storage.

C. Storage:

1. Refrigeration: When samples are not in use, that is during sample log in, sample preparation, etc. they must be kept refrigerated in the central laboratory cooler at 1 - 4 degrees centigrade.

D. Holding Times

1. Extraction: Aqueous samples must be extracted within 7 days of sample collection. Soil samples must be extracted within 14 days of sample collection.
2. Analysis: following extraction, both aqueous and solid samples have a 40 day holding time from the time of extraction until analysis.

VII. SAFETY

A. General Precautions: Many of the semi-volatile analytes are either suspected or known carcinogens and must be treated with great care. Analysts should handle these chemicals with Nitrile gloves and with proper ventilation. Skin contact should be avoided as well as inhalation.

1. Laboratory: When extracting samples, protective eye glasses must be worn at all times and methylene chloride vapors must be ventilated properly. Ventilating separatory funnels should be done under the canopy hoods, while KD concentration should be performed under the sash hoods. Analyst should where gloves and a lab coat when performing these operations.
2. Chemicals: Solvents should be stored in the ventilated explosion proof cabinet located in the extraction lab. Additional solvent is kept in the garage. Standards should be stored in the freezer to minimize degradation. Follow manufacturers recommendations if storage instructions differ.
3. Personal: Wear eye glasses, gloves and lab coat when performing sample extractions. Loading the instrument requires eye protection.
4. MSDS information is kept on file and the analyst is expected to be familiar with necessary precautions.

VIII. EQUIPMENT AND MATERIALS

A. Instrumentation: GC-MSD system is by Hewlett-Packard, model 5972. The GC is the Hewlett-Packard model 5890, series II with electronic pressure control. Computer data station is the HP Vectra XM2/100i with the Ultra VGA 1280 monitor. Printer is the HP Laser Jet 4 Plus. The Rough Pump is the Edwards RV3 model. System includes the ChemStation and EnviroQuant Software for instrument control data acquisition and reduction.

B. Extraction/Preparation: PFTE boiling chips, water bath with heater control and concentric ring cover.

C. Glassware

1. Specification: 2 Liters separatory funnels with Teflon stopcock, 500 mL KD evaporation flask, 10 mL concentrator tubes, three ball Snyder column, 2 mL glass vials, 1 Liter graduated cylinder.
2. Preparation: All glassware (except 3 ball Snyder columns) must be washed in hot soapy water with Laboratory Detergent Concentrate, followed with a final cold water rinse. Glassware is then rinsed with methanol to remove any water. Allow glassware to drip dry on the glassware racks in the extraction lab.

IX. REAGENTS AND STANDARDS

A. Reagent Purity Specifications

1. Analytical standards should be purchased from a reputable supplier (See Section XVIII Part C of this document). The certificate of analysis sheets must be kept on file so that all standards have a well documented paper trail.

B. Standards Preparation Directions

1. Initial Calibration Curve Setup: Prepare 5 mL of the nominal 80 ug/mL standard with the following Standards. This is for the typical NLS 8270C method.

	Inject (uL)	Ultra Mix
Restek		
Restek	200	Mix 1 @ 2000 ug/ml
Restek	200	Mix 2 @ 2000 ug/ml
Restek	200	Mix 3 @ 2000 ug/ml
Restek	200	Mix 4 @ 2000 ug/ml
Restek	200	Mix 5 @ 2000 ug/ml
Restek	200	Mix 6 @ 2000 ug/ml
Restek	200	Appendix IX custom @ 2000
Protocol	200	Custom NLS Vol. 7
NSI	800	Q Surr 2052

a. Next perform the serial dilution off the 80 ug/mL into 6 additional levels as follows:

Calibration Level ug/mL	Amount of 80 std (uL)	Amount of Methylene Chloride (uL)
50	125	25
40	100	100
30	75	125
20	50	150
10	25	175
5	12.5	187.5

Add 2 uL of Internal standard to each 200 uL volume Solution. Standards are now ready for injection. Internal standard is @ 4000 ug/ml.

2. Continuing Calibration Check Standard is used to validate the initial calibration curve. Final standard concentration is 40 ng/uL. Perform a 2X serial dilution from the 80 ug/ml standard.

3. Miscellaneous Standard Preparation:

a. All 1.0 ml samples receive 10 uL of internal standard. (One uL of IS per 100 uL of sample.) Final Concentration of IS in solution is 40 ng/uL.

b. DFTPP Tune Standard (1,000 ug/mL) is prepared with 10 uL of the tune mix, plus 190 uL of DCM. Final Concentration of DFTPP in solution is 50 ng/uL.

c. Surrogate Spike Mix use .5 ml of the NSI BNA Surrogate mixture which contains both Acid & Base compounds.

d. MS/MSD and LCS use .5 ml TCL BNA Spike w/o Benzidines.

C. Storage Conditions: Most standards should be stored in the freezer and kept out of the light to prevent photochemical decomposition.

D. Shelf life: Typical shelf life for most stock Semi-volatile standards is one year. With typical usage, solutions will normally be used up within a year. Standards are dated upon receipt, and discarded before one year.

X. PROCEDURE

A. Extraction Preparation

1. Liquid Samples

a. For liquid samples measure approximately 1 liter of the sample and transfer it quantitatively to the separatory funnel. Record this volume in the Sample Preparation Logbook. If high concentrations are anticipated, a smaller volume may be used and then diluted with organic free water to 1 liter. Add .5 ml of the surrogate standard to all samples, blanks and spikes. The resultant concentration for the base-neutral compounds should be 50 ug/L while the final concentration for the acid extractable surrogate compounds should be 100 ug/L. For the sample in each analytical batch selected for the spikes (MS & MSD), add .5 ml of thespiking standard mix.

b. Check the pH of the sample prior to extraction and adjust the pH according to the following determinative method: Acid extractables require pH < 2, while Basic extractables require pH 11. Acidic conditions are created by adding 50 ml of 1:1 sulfuric acid:water solution. Basic conditions are created using 10 N NaOH solution. The current pH sequence is neutral, base then acid, with three extractions for each pH.

c. Add 50 mLs of Methylene Chloride to the separatory funnel. Seal and shake the separatory funnel vigorously for 1 - 2 minutes with periodic venting (under the canopy hood) to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of10 minutes. If an emulsion interface between the layers forms and is more than one-third thesize of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. These techniques may include stirring, filtration through glass wool, centrifugation, or other physical methods. Drain the solvent into a KD boiling flask through 10 cm of anhydrous sodium sulfate.

d. Repeat the procedure two more times with 60 ml portions of Methylene Chloride.

2. Solid Samples:

a. For solid samples (see SW-846 method 3550B) the procedure is as follows:

b. Weigh approximately 30 g of sample into a 400 ml beaker. Record the weight to the nearest 0.1 g. Nonporous or wet samples (gummy or clay type) that do not have a free-flowing sandy texture must be mixed with 15 g of anhydrous sodium sulfate, using a spatula. If required, more sodium sulfate, may be added. After addition of sodium sulfate the sample should be free flowing. Add .5 ml of surrogate solution to all samples, spikes and blanks. For the sample selected for the MS and MSD add .5 ml of the spiking solution (see section IX, part B (3d)). Immediately add 50 ml of Methylene Chloride.

c. Next place the bottom surface of the tip of a 3/4 inch disrupter horn about 1/2 inch below the surface of the solvent but above the sediment layer. Extract ultrasonically for 3 minutes, with output knob at 10 (full power) and with mode switch on Pulse and percent duty knob set at 50% energy. Do not use microtip probes.

d. Decant the extract and filter it through Silanized glass wool with a 2 inch layer of sodium sulfate in a funnel that is attached to a clean concentrator flask. Repeat the procedure two more times with 50 ml portions of solvent. On the final ultrasonic extraction, pour the entire sample into the funnel and rinse with the extraction solvent. Sample extract is now ready for concentration.

3. Sludge Samples:

a. For liquid samples with 30 to 2 percent solids use the continuos liquid-liquid extraction procedure method 3520C, NLS SOP Number ORG-METH-EXTR-L/L Continious-0.
b. Weigh approximately enough material to yield 10 grams of sample DWB. Pour the sample into a graduated cylinder and record the volume. Fill to 1 Liter with organic free water.
c Fill the bottom of the extractor with about 2 1/2 inches of Methylene Chloride. Slowly pour the liquid sample into the extractor. The Methylene Chloride layer will be on the bottom and some of the solvent will be forced up the glass tube under the weight of the sample.
d. Fill the round bottom flask with approximately 250 mls of Methylene Chloride and add the
e. boiling stones. Next attach the round bottom flask to the extractor placing the flask into the heating mantel.
f. Spike all the samples with .5 ml of the surrogate solution and the MS/MSD and LCS with .5 mls of the spike solution. The acid surrogate concentration should be at 100 ug/L and the neutral surrogate should be at 50 ug/L. The matrix spike concentration should be at 50 ug/L.
g. Acidify the samples with 50 ml of 1:1 Sulfuric Acid:Water to a pH < 2. Connect the water condenser to the top of the extractors and secure the apparatus. Open the water valve and set the heating level at 50. Extract for 18-24 hours.
h. Turn down the heat and let the samples cool. Raise the sample pH to > 11 with 10N NaOH. Set the heating level to 50 and extract for 18-24 hours.
i. Pour the solvent into a K-D flask through approximately 20 grams of Sodium Sulfate (in a funnel) to dry the extract. Sample is now ready for concentration.

B. Final sample preparation

1. Concentration Specifications: Add several PFTE boiling chips to the KD flask. Place the KD flask into the water bath at a temperature of 75 to 85 degrees F. Gently boil the solvent so that the balls "chatter" in the Snyder chamber without flooding the chamber with solvent. Remove the KD flask when the solvent reaches 1 ml. Allow the flask to cool and drain back into the concentrator tube.
   a. If further concentration is needed do not use the Nitrogen Blowdown Technique as described in method 3510B. This technique will adversely effect spike recovery for the low boiling analytes. The Micro-Snyder Column technique is an alternate way to further concentrate (1 ml or less) the samples without loss of analytes. This technique involves the use of a small boiling flask (50 ml) connected to the same concentrator tube topped of with the micro-Snyder column.
   b. Concentrate to a final volume of 1 ml of solvent in the concentrator tube and record this volume in the Sample Extraction Logbook.
2. Dilution requirements: Sometimes a sample exhibits characteristics (strong petroleum odor & dark color) which suggest a dilution prior to analysis is advisable. If a sample requires dilution, simply remove an aliquot (with a syringe) of the final extract and dilute up to 1 ml with Methylene Chloride in an autosampler vial. Record the dilution factor in the Sample Extraction Logbook and the Instrument Run Logbook. Store the undiluted extract in the freezer in the event the sample needs to be reanalyzed at a different dilution.

XI. INSTRUMENT ANALYSIS

A. GC/MS operating conditions:

Mass Range:	35 - 500 amu
Scan Time:	1.4 scan/sec
Initial Temperature:	40 degree C, hold 1 minutes
Temperature Program:	Ramp 35 C/min to 130, hold 0 minutes
Final Temperature:	Ramp 12 c/min to 325, hold 7 minutes
Run Time:	26.82 minutes
Injector Temperature:	250 degrees C
Source Temperature:	280 degrees C
Injector:	Split/Splitless
Sample Volume:	1 uL injection
Carrier Gas:	Helium at 36 cm/sec - EPC flow 1.3 ml/minute
Electron Multiplier:	minute solvent delay

The GC oven temperature program is a suggested program and will typically be used for most analysis. The mass spec settings should not be altered.

B. Tuning the MSD

1. The GC/MS must pass the tune specifications listed below by injecting 50 ng of DFTPP (decafluorotriphenylphosphine).

Mass	Ion Abundance Criteria
51	30-60 % of mass 198
68	< 2 % of mass 69
70	< 2 % of mass 69
127	40 - 60% of mass 198
197	< 1 % of mass 198
198	Base Peak, 100 % relative abundance
199	5 - 9% of mass 198
275	10 - 30 % of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40 % of mass 198
443	7 - 23 % of mass 442

2. This criteria must be demonstrated each 12 hour shift. In other words if the tune passes you may proceed with the analysis for up to 12 hours. The last accepted sample must be injected within 12 hours of the time posted on the DFTPP report. All methods should use the DFTPP.U file as the tune file. If the tune fails repeatedly with a fresh DFTPP standard the target tune relative abundances may have to be changed or the source may be getting dirty and it is time for cleaning.

3. To reset the target tune abundances use the following procedure. Under MSTOP menu go into TUNEMS. Click on TARGET TUNE. Select TUNE. Then select LOAD TUNE VALUES and select the DFTPP.U file. Next, select SET TUNE. Load Tune Targets DFTPP.TGT Enter in a relative abundance for the 131 Ion, 219 Ion and the 502 Ion. Once these abundances are set select SAVE TUNE TARGETS to DFTPP.TGT. Now select DFTPP TUNE with these new values. The mass spec will go through its tune routine and generate a report. Before exiting TUNE, save the tune values just established under file, save Tune values; DFTPP.U. Now reinject the DFTPP

standard under these new settings to see if it will meet the necessary tune specifications. If the tune still does not pass try tuning again with different target tune abundances.

Some suggested settings for the relative abundance:

ION	% RELATIVE ABUNDANCE
131	24, or 25
219	26, or 27
502	0.8

4. If after several iterations, the DFTPP standard will still not pass it may be time to replace the injection port liner and gold seal or the Ion Source may need cleaning. Before cleaning the source, reference HP 5972A MSD Hardware Manual and watch the video "Cleaning the HP 5972A Ion Source".

C. Calibration:

1. Method 8270C requires a five point calibration curve. In order to meet the most stringent requirements, the calibration curve should consist of 6 calibration points with the lowest concentration near the detection limit.

2. There are four compounds called System Performance Check Compounds (SPCCs). These compounds must have a minimum average response factor greater than 0.05. These compounds tend to decrease in sensitivity as the chromatographic system deteriorates or the standard material degrades. If the minimum response factors are not met the system must be evaluated, and corrective action must take place before sample analysis begins. The four SPCC compounds are:

N-nitroso-di-n-propylmaine
Hexachlorocyclopentadiene
2,4-Dinitro-phenol
4-Nitrophenol

3. Once the SPCC compounds have met specification of the thirteen Calibration Check Compounds (CCCs) must be evaluated. The percent relative standard deviation % RSD of the relative response factor of the five calibration points must be less than 30% for each CCC. The CCC compounds are:

Acenapthene
1,4-Dichlorobenzene
Hexachlorobutadiene
N-Nitrosodiphenylmaine
Di-n-octyl phthalate
Fluoranthene
Benzo (a) pyrene
4-Chloro-3-methylphenol
2,4-Dichlorophenol
2-Nitrophenol
Phenol
Pentachlorophenol
2,4,6-Trichlorophenol

4. All other compounds should have an RF %RSD less than 15 %. If the %RSD of any compound is 15 % or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation. If the %RSD of any compound is greater than 15 %, a first or higher order regression fit of the five calibration points may be used for A/A_{is}) vs concentration. This is a recommended alternative to average response factor calibration.
5. Prior to sample analysis the initial calibration must be validated with a continuing calibration standard (CCAL). The RF of the CCCs for the CCAL must be less then 20 percent difference of the average RF of the ICAL. The SPCCs must have an RF > 0.05.
6. Following the CCAL, analyze a method blank or solvent blank prior to sample analysis to demonstrate that the system is free from carry-over.

D. Sample/Standard Presentation to Instrument

1. All sample extracts must be delivered to the 1.8 ml glass autosampler vials and labeled with a complete description. All samples must be injected with 1 uL of the 4000 ug/mL internal standard mix for every 100 uLs of sample. For example, 1.0 ml of sample would receive 10 ul of the 4000 ug/mL internal standard mix for a final sample concentration of 40 ug/mL.

Internal Standard Compounds

Acenaphthene-d10
Chrysene-d12
1,4-Dichlorobenzene-d4
Naphthalene-d8
Perylene-d12
Phenathrene-d10

2. The resultant sample/extract concentration is 40 ug/mL for the internal standard compounds.

XII. CALCULATIONS

A. General Calculations

1. Response Factor:

$$RF = (A_s C_{is})/(A_{is} C_s)$$

Where: A_s = Area response for the analyte's Quant Ion
C_{is} = Concentration of the internal standard (ug/L)
A_{is} = Area response for the internal standard Quant Ion
C_s = Concentration of the analyte (ug/L)

2. Final Extract Concentration:

$$Cex = (A_x C_{is})/(A_{is} RF_{avg})$$

Where: A_s = Area response for the analyte's Quant Ion
C_{is} = Concentration of the internal standard (ug/L)
A_{is} = Area response for the internal standard Quant Ion
RF_{avg} = Average response factor of the initial calibration
C_{ex} = Final concentration in the extract (ug/ml)

3. Final Liquid Concentration:

$$Cf = (D_1 C_{ex} V_{ex})/Vi$$

Where: C_f = Final concentration in the sample (ug/L)
D_1 = Dilution of the extract (unitless)
C_{ex} = Final concentration in the extract (ug/ml)
V_{ex} = Final volume of extract (ml)
V_i = Initial volume of the sample (L)

4. Final Soil Concentration:

$$C_f = (D_1 C_{ex} V_{ex})/(M_i S)$$

Where: C_f = Final concentration in the sample (ug/Kg DWB)
D_1 = Dilution of the extract (unitless)
C_{ex} = Final concentration in the extract (ug/ml)
V_{ex} = Final volume of extract (ml)
M_i = Initial Mass of the sample (L)
S = Decimal equivalent of the percent solid content of the soil

B. Significant figures:

1. Sample results should be reported to two significant figures except for all Blind and Performance Evaluation studies which require three significant figures. The results entered into the template will automatically be rounded to the specified number in the reporting template.

C. Special Adjustments for Samples:

1. Sample Size: Typical liquid sample volume is 1 Liter. Smaller volumes may be used if necessary but this will elevate the final sample detection limit. Typical soil size is 30 grams, however a smaller size is permissible if desired. Smaller sample size will elevate the final detection limit.
2. Sample Matrix: If the matrix is an oily liquid employ the waste dilution procedure in accordance with EPA 3580.

XIII. QUALITY CONTROL

A. Standards:

1. Standards should be purchased from a reliable supplier (some listed on last page) and accompanied with a certificate of analysis which verifies the sample concentration for a specific lot number. The certificate of analysis must be kept in a file for a complete record of the standard preparation. Lot numbers and labels should be recorded and attached in the Standard Preparation Logbook. When standards are prepared from a stock standard they must be labeled properly with the date, concentration and description of the mix.

B. Lab Control Standard:

1. Laboratory Quality Control Standard (LCS) is called a method performance verification standard and is used to determine the extraction efficiency of various compounds for a given extraction batch. The method performance verification standard ensures that the process prior to analysis are functioning properly and are producing their intended result. An LCS is required by the method and is a good indicator of extraction performance apart from any matrix affects. An LCS is a very useful tool in troubleshooting the method.

2. Both the MS/MSD are samples (soil or water) spiked with a known amount of analyte and extracted along with the other samples within the extraction batch. The result of the MS and MSD provide a measure of the accuracy and precision of the extraction batch. The accuracy is expressed as percent recovery and the precision is expressed as percent difference. The matrix performance verification standard is called a Matrix Spike (MS) or Matrix Spike Duplicate (MSD).

3. An extraction batch is defined as twenty sample extractions and should always have one Blank, MS, MSD and LCS. In cases where there is not enough sample available to perform an MS/MSD an LCS and LCS duplicate should be substituted.

C. Sample QC

1. Accuracy measurements (spike percent recovery):
   a. QC spike acceptance criteria is defined by the upper and lower control limits established by the MS/MSD. Once a minimum of 30 sample points are collected the acceptance range is determined as follows: (p: percent recovery, s_p: standard deviation of percent recovery).

$$\text{Upper Control Limit} = p + 2s_p$$
$$\text{Lower Control Limit} = p - 2s_p$$

2. Precision measurements (duplicate - relative percent difference)
3. Surrogate measurements (percent recovery range)
   a. Surrogate spike acceptance criteria is defined by the upper and lower control limits established by the samples analyzed. Once a minimum of 30 sample points are collected the acceptance interval is determined as follows: (p: percent recovery, s_p: standard deviation of percent recovery).

$$\text{Upper Control Limit} = p + 3s_p$$
$$\text{Lower Control Limit} = p - 3s_p$$

D. Analytical Limitations:

1. Sample Interference: Phthalate Esters are the most common interferent and are associated with plastics. Other sources of artifacts may be the reagents, standards and glassware. Use high purity gasses and reagents and be careful to rinse glassware properly to avoid sample contamination. Also it is important to use good laboratory technique when preparing standards or samples in order to minimize introduction of unwanted artifacts. Syringes should be triple rinsed after exposure to standards or sample concentrates.
2. Instrument Limitations: Lower quantitative limit is defined by the LOQ. Analytes may be detected at or near the MDL and should be considered the lowest detectable concentration. The upper quantitative limit is defined by the highest calibration standard in the initial calibration. Estimated sample concentrations may be determined above the highest point on the curve but should only be used to gauge the necessary dilution for the re-analysis. Qualitative limitations are defined by the temperature range of Semi-Volatile compounds, the calibration curve and the NIST library.

E. Method Validation: Each analyst performing this analysis must successfully complete the initial demonstration of proficiency requirement stated in the method. This involves the extraction and analysis of 4 reagent water samples spiked with a known amount of analyte. The final numbers should be compared to the accuracy and precision tables in the method to determine if the analyst has successfully met the minimum performance criteria. In addition the laboratory QA officer will issue Blind Performance Evaluation samples to test the analyst's proficiency. These Blind studies usually coincide with State and Federal Performance Evaluation studies conducted periodically for various monitoring programs.

F. MDL study procedure: Each analyte reported should have a Method Detection Limit (MDL) statistically computed according to 40 CFR, Part 136, Apdx B, Rev 1.1. MDLs should be performed at least yearly or whenever significant changes are made to the instrument impacting MSD sensitivity.

XIV. RECORDS AND REPORTING DATA

A. Benchsheet entries: See attachment VI for an example of the benchsheet used. Benchsheets serve only as a work order, and are not kept on file.

B. Units/Significant figures

1. Liquid samples are reported as ug/L at 2 significant figures.
2. Solid samples are reported as ug/Kg DWB at 2 significant figures.

C. Detection limits and Reporting limits: All detection limits are determined according to 40 CFR, Part 136, Apdx B, Rev 1.1. If the MDL is not determined by this procedure for a given compound a conservative estimate may be used in the interim. This estimate should be noted on the final report. For additional references on MDL determination see Analytical Detection Limit Guidance, WDNR OTS, April 1995 draft. See attachment II for a complete listing of individual MDLs and LOQs.

D. Qualifiers or comments used if data is to be flagged:

1. Samples must be flagged with a specific comment if any of the following situations arise and are not corrected through a sample rerun, re-extraction or recalibration.
   a. Contaminated Extraction Blank.
   b. Surrogate Recovery out of range.
   c. Holding time exceeded.
   d. MS/MSD spike recovery exceedance.
   e. Continuing Calibration out of range.
   e. CCC & SPCC compounds outside tolerance.

E. LIMS Entry

1. The laboratory information system contains all of the data which appears on the final analytical reports. Results are entered into the LIMS system under template entry. Simply enter the appropriate test code, initials under who, date and time and dilution. In result entry, enter "see attached". If there are any footnotes, comments or qualifiers enter this information under the notes category.

F. Client Reports:

1. After all the data is entered, return to the previous menu and print results on the final report.

G. Data archiving or filing:

1. Archiving of HP-MSD should be performed on a regular three-month basis. If the hard drive fills up while acquiring data, it will lock up. If this happens it may be necessary to reload all of the software. Make certain that there is enough space for data acquisition. Data files are transferred to the LIMS archive.

H. Hard Copy Archiving: Processed data reports are printed for each sample analyzed. A copy of the final report should be stapled to the processed report. All data compiled from an instrument run should be kept together in the data package. Information kept in the file folder includes, the DFTPP Tune report, Continuing Calibration Summary, MS & MSD reports (if analyzed), extraction and instrument blanks, and all sample data. The data package should be labeled with the date of the analytical run and stored in the vertical file cabinet located in the laboratories central office area. Periodically, data will be moved to the basement archives as the file cabinet fills up.

I. Instrument Run Logbook: See attachment III for an example of the instrument run logbook. The run logbooks must be filled out for each analytical run.

J. Standard Preparation Logbook: See attachment V for an example of the Standard Preparation Logbook. The standard preparation logbook is filled out every time a new standard solution is opened or prepared. If an adhesive label is available it should be attached to the page containing the information about the standard prepared. This label is then traceable to the certificate of analysis for a complete record.

K. Maintenance Logbook: The maintenance logbook is a ring binder notebook with 5 divisions: INT STD contains internal standard information, MAINTENANCE contains the record of all maintenance activities, INTEGRATION contains specific peak integration instructions, FILES contains all of the miscellaneous file names and their descriptions, 502 ION contains a running record of the 502 abundance and EM voltages for various tunes. This information should be kept up to date for an accurate record of the instrument performance overtime.

XV. CLEAN UP

A. Lab Work Area: Samples should always be returned to the walk-in cooler after use. Also, any materials, reagents, supplies or standards used throughout the day should be returned to their proper location. The lab area in general should always be kept clean and free from clutter.

B. Sample Disposal & Standard Disposal: Samples are disposed of in accordance with procedures determined in the QA/QC manual. Soil samples are discarded into a soil waste drum. Liquid samples are emptied down the drain and the glass amber containers are placed in a storage area in the old lab. Glass amber bottles are not reused. Standards and sample extracts are emptied from the 2 ml vials into a solvent waste jug designated for GC/MS Semi-Volatile samples. The glass vials are discarded into a glass jug that is later disposed. The solvent and standard contents will be removed from the premises by a waste hauler.

C. Equipment/Glassware: Equipment should be kept clean and dust free. Occasional cleaning may be needed to ensure equipment is free from dust and other debris. Also, glassware should be cleaned as soon as possible after use. If immediate cleaning is not possible, all used glassware should be left to soak in a tub of hot soapy water until it can be cleaned.

XVI. MAINTENANCE/TROUBLESHOOTING

A. Preventive maintenance procedures and frequency

1. Check daily the vacuum manifold pressure - Should read about 7.5 x 10^{-5} TORR.
2. Check weekly the Rough Pump Oil Level.
3. Check weekly the pressure of the Carrier Gas (He).
4. Check monthly and refill the CAL gas vial as necessary.
5. Replace every three months the Rough Pump Oil.
6. Replace every six months the Rough Pump Trap Pellets.
7. Check yearly and replace if necessary the Diffusion Pump oil.
8. As needed clean the Ion Source.
9. As needed check the oxygen Carrier Gas Trap and replace.
10. As needed replace the Filament and Multiplier.
11. As needed (100 injections) replace the injector septa.
12. As needed replace injection port glass insert & 0-ring, gold seal & washer.

B. Troubleshooting procedures: See Chapter 3 of the Hewlett-Packard 5972A Hardware Manual.

XVII. ATTACHMENTS

A. Attachment II: Method detection limits and Limits of quantitation.

B. Attachment III: Instrument Run Logbook.

C. Attachment IV: Sample Preparation Logbook.

D. Attachment V: Standard Preparation Logbook.

E. Attachment VI: Benchsheets.

XVIII. MISCELLANEOUS INFORMATION

A. Service Phone Numbers:

1. For questions on part numbers & pricing for HP supplies call (916) 783-0804.
2. For technical questions as to what the parts do and what parts are necessary for HP systems call (800) 424-9759.
3. General HP number (800) 227-9770 Purchases, etc.

4. For Technical Support/Service Calls contact LINC Quantum Analytics (800) 458-9641 or AIS (Analytical Instrumentation Service, Inc.) (708) 739-4135, ask for Greg Bedenk.

B. Special HP Part Numbers

1. Injector Inserts: 5062-3587, these are pre-cleaned and deactivated.
2. On-column Needles: 5182-0832, these needles attach to the syringe barrel and are used for automatic on-column injections. A 0.530 mm pre-column must be joined to the 0.25 mm ID capillary column.
3. Ferrule for Injector: 5181-3323, these 10% graphite, 90% Vespel®, and are for 0.25 mm column ID, comes as a package of 10.
4. Ferrule for Detector Inlet: 5062-3508, these are 15% graphite, 85% Vespel®, and are for 0.25 mm Column ID, comes as a package of 10.
5. Gold Plated Seal for Injector: 18740 - 20885.
6. Washer for Injector (12/pk): 5061-5869.

C. Standard Supplier Telephone Numbers:

1. Ultra Scientific: (800) 338-1754
2. NSI:(800) 234-7837
3. ChemService:(800) 452-9994
4. Restek:(800) 356-1688

D. Equipment Model and Serial Numbers: Installation & Warranty Start Date: 08/08/95.

Model #	Serial Number	Description
5972A	3501A02657	Mass Spec
589OE	3336A59178	Gas Chromatograph
D3233A	3318A66831	Vectra Xm2 4/100i
D2807A	JP50807564	Ultra VGA 1280 Monitor
C2037A	JPGL072537	LaserJet 4 Plus
G1512A	3529A02405	ALS Controller
18596M	3522A38797	ALS Tray
18593B	3529A43280	ALS Tower
59864A	342010	ION Gauge Controller
RV3	03594	Rough Pump
G1033A	3235A23472	Environquant Software
G1034C	N/A	MS Software

NORTHERN LAKE SERVICE

HP 5890 GC / HP 5972 MSD - GC/MS INSTRUMENT RUN LOG

8270 SEMI-VOLATILES ANALYSIS LOGBOOK

BOOK: _____

PAGE: _____

Date: / /	Method: 8270 -	Analyst:	Calibration Date: / /

File No.	Sample Number	Initial Vol./Mass	Final Volume	Dil. Fact.	2-Fluorophenol (S.S.) [% Recovery]	Phenol-d5 (S.S.) [% Recovery]	Nitrobenzene-d5 (S.S.) [% Recovery]	2-Fluorobiphenyl (S.S.) [% Recovery]	2,4,6-Tribromophenol (S.S.) [% Recovery]	Terphenyl-d14 (S.S.) [% Recovery]	Comments
1	DFTPP										
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											
15											
16											

General Comments:	Internal Standards	Area Counts Must Be -50% to + 100% CCAL		Standards Reference	Book #	Page #	Line #
		Initial Calibration Area Counts	Continuing Calibration Area Counts				
				DFTPP Mix:			
	1,4-Dichlorobenzene–d4			Internal Standard Mix:			
	Naphthalene-d8			8270 Calibration Mix:			
	Acenaphthalene-d10						
	Phenanthrene-d10						
Project Numbers:	Chrysene-d12						
Run Time: To:	Perylene-d12						

NORTHERN LAKE SERVICE, INC.
GC/MS SEMI-VOLATILES
SAMPLE PREPARATION LOGBOOK

BOOK: _____
PAGE: _____

Date:	Extr.Method (*):	Surr. Mix:	CH_2Cl_2 Lot:	(10N) NaOH Lot:
Analyst:	Extr. Batch:	Spike Mix:	$NaSO_4$ Lot:	(1:1) H_2SO_4 Lot:

Line No.	NLS Sample Identification	INITIAL Mass / Volume	FINAL Volume	Surrogate Volume	Spike Volume	Matrix Type (Ψ)	(N) pH_1	(B) pH_2	(A) pH_3	Comments
1	BLK									
2	MS									
3	MSD									
4	LCS									
5										
6										
7										
8										
9										
10										
11										
12										
13										
14										
15										
16										
17										
18										
19										
20										

(*) Methods: 3510B = Liq/Liq; 3550A = Sonication; 3580 = Waste Dilution; 3535 = SPE

(Ψ) Matrix Type: S = Solid; TW = Tap Water; W = Wastewater; DW = Drinking Water; C = Clean; OS = Ottawa Sand; SL = Sludge

KEY	BLK = Blank	MS = Matrix Spike	MSD = Matrix Spike Duplicate	LCS = Lab Control Spike	A = Acid	B = Base	N = Neutral

NORTHERN LAKE SERVICE, INC.
GC/MS SEMI-VOLATILES
STANDARDS PREPARATION LOGBOOK

BOOK: _____
PAGE: _____

Line No.	Date	Vendor	Compound / Mixture	Lot #	Exp. Date	Stock Conc.	Dil / Prep	Solvent Lot #	Final Conc.	Mix Ref Number	Analyst	Discard Date	Discard By
1													
2													
3													
4													
5													
6													
7													
8													
9													
10													
11													
12													
13													
14													
15													
16													
17													
18													
19													
20													

Solvent is Dichloromethane unless specified

Comments

List

NAME	ESORT	PEAK	LOD	LOQ	UNITS	PCOD	RSOR
8270WX	1	Pyridine	.77067022	2.568907	ug/L	77045	99.1
8270WX	2	2-Picoline	.9632006	3.210669	ug/L	77088	97
8270WX	3	n-Nitrosomethylethylamine	1.288897	4.296323	ug/L	73613	116
8270WX	4	Ethyl Methanesulfonate	.6004151	2.001384	ug/L	73571	59
8270WX	5	n-Nitrosodimethylamine	1.363781	4.545936	ug/L	34438	78
8270WX	6	Methyl Methanesulfonate	.8535796	2.845266	ug/L	73595	72
8270WX	7	Aniline	1.34804	1.34804	ug/L	77089	6
8270WX	10	Phenol	.5070925	1.690308	ug/L	34694	95
8270WX	11	n-Nitrosodiethylamine	1.106454	3.688181	ug/L	78200	114
8270WX	12	Bis(2-chloroethyl)ether	.8059323	2.686441	ug/L	34273	18
8270WX	13	2-Chlorophenol	.8976755	2.992252	ug/L	34586	26
8270WX	14	1,3-Dichlorobenzene	.5303755	1.767918	ug/L	34566	37
8270WX	15	1,4-Dichlorobenzene	.5302543	1.767514	ug/L	34571	36
8270WX	16	1,2-Dichlorobenzene	.4978651	1.65955	ug/L	34536	35
8270WX	17	Benzyl Alcohol	.7788041	2.596014	ug/L	77147	17
8270WX	18	Bis(2-chloroisopropyl)ether	.8700739	2.900246	ug/L	73522	111
8270WX	19	2-Methylphenol	1.208514	4.028381	ug/L	77152	74
8270WX	20	n-Nitrosopyrrolidine	1.246245	4.15415	ug/L	78206	118
8270WX	21	n-Nitrosomorpholine	.81939	2.7313	ug/L	73617	117
8270WX	22	0-Toluidine	1.528529	5.095096	ug/L	77142	121
8270WX	23	Hexachloroethane	.8293716	2.764572	ug/L	34396	65
8270WX	24	n-Nitroso-di-n-propylamine	1.045821	3.486069	ug/L	34428	76
8270WX	25	Acetophenone	.8851544	2.950515	ug/L	81553	3
8270WX	27	3 & 4-Methylphenol	1.266717	4.222389	ug/L	77146	75
8270WX	29	O,O,O-triethyl Phosphorothioate	.9878467	3.292822	ug/L	73652	124
8270WX	31	n-Nitrosopiperidine	1.154123	3.847077	ug/L	73619	79
8270WX	32	n-Nitrosodi-n-butylamine	1.048399	3.494664	ug/L	78207	77
8270WX	33	2,4-Dichlorophenol	1.144327	3.814423	ug/L	34601	40
8270WX	35	Nitrobenzene	.9986553	3.32851	ug/L	34447	86
8270WX	36	Isophorone	1.042288	3.474293	ug/L	34408	69
8270WX	37	2-Nitrophenol	1.605766	5.352552	ug/L	34591	87
8270WX	38	2,4-Dimethylphenol	1.60971	5.365698	ug/L	34606	45
8270WX	39	Bis(2-chloroethoxy)methane	1.084603	3.615344	ug/L	34278	109
8270WX	40	2,6-Dichlorophenol	1.149987	3.833289	ug/L	77541	39
8270WX	41	1,2,4-Trichlorobenzene	.521591	1.738637	ug/L	34551	105
8270WX	42	Naphthalene	.734661	2.44887	ug/L	34696	80
8270WX	43	4-Chloroaniline	1.043852	3.479506	ug/L	73529	22
8270WX	44	Hexachloropropene	.4506959	1.50232	ug/L	73576	66
8270WX	45	Hexachlorobutadiene	.4924121	1.641374	ug/L	34391	63
8270WX	46	a,a-Dimethylphenethylamine	.5725881	1.908267	ug/L	73564	108
8270WX	47	p-Phenylenediamine	30	90	ug/L	73628	123
8270WX	48	4-Chloro-3-methylphenol	1.2060385	4.201283	ug/L	34452	21
8270WX	49	Cis-Isosafrole	.6778274	2.259425	ug/L	73582	29
8270WX	50	Safrole	.8025851	2.675283	ug/L	77545	100
8270WX	51	2-Methylnaphthalene	.7646428	2.548809	ug/L	77416	73
8270WX	53	Hexachlorocyclopentadiene	1.06	3.37	ug/L	34386	64
8270WX	54	1,2,4,5-Tetrachlorobenzene	.70897781	2.36326	ug/L	77734	102
8270WX	55	1-Chloronaphthalene	.95339	3.036288	ug/L	38687	24
8270WX	56	4-Nitrophenol	.43	1.43	ug/L	34646	88
8270WX	57	Pentachlorobenzene	.5356371	1.788[illegible]7	ug/L	77793	99

NAME	ESORT	PEAK List	LOD	LOQ	UNITS	PCOD	RSOR
8270WX	58	1-Naphthylamine	4.58	15.2	ug/L	73600	82
8270WX	59	2-Naphthylamine	3	9.57	ug/L	73601	81
8270WX	60	2,3,4,6-Tetrachlorophenol	.6168526	2.056175	ug/L	77770	103
8270WX	61	2,4,6-Trichlorophenol	.7903875	2.634625	ug/L	34621	106
8270WX	62	2,4,5-Trichlorophenol	1.041548	3.471825	ug/L	77687	107
8270WX	64	2-Chloronaphthalene	1.35	4.5	ug/L	34581	25
8270WX	65	2-Nitroaniline	.7813176	2.604392	ug/L	78142	84
8270WX	66	Acenaphthylene	1.29	4.3	ug/L	34200	2
8270WX	67	1,4-Naphthoquinone	4.94	2.774855	ug/L	73599	119
8270WX	68	1,4-Dinitrobenzene	.8324566	2.774855	ug/L	99149	135
8270WX	69	1,2-Dinitrobenzene	.4629	1.543	ug/L	99148	135
8270WX	70	Dimethylphthalate	.94113965	3.137988	ug/L	34341	46
8270WX	71	1,3-Dinitrobenzene	.6326925	2.014944	ug/L	45622	48
8270WX	72	2,6-Dinitrotoluene	.6137705	2.045902	ug/L	34626	52
8270WX	73	Acenaphthene	.6683121	2.22707	ug/L	34205	1
8270WX	74	3-Nitroaniline	.8741925	2.913975	ug/L	78300	83
8270WX	75	2,4-Dinitrophenol	.6197638	2.065779	ug/L	34616	50
8270WX	76	Dibenzofuran	1.32	4.4	ug/L	81302	34
8270WX	77	2,4-Dinitrotoluene	.6371911	2.123971	ug/L	34611	51
8270WX	78	Fluorene	.6085111	2.02837	ug/L	34381	61
8270WX	79	4-Chlorophenyl-phenylether	.5074569	1.691523	ug/L	34641	27
8270WX	80	Diethylphthalate	1.4	4.65	ug/L	34336	41
8270WX	81	Thionazin	.9648094	3.216032	ug/L	73553	104
8270WX	82	5-nitro-o-toluidine	.7212377	2.404126	ug/L	73622	120
8270WX	83	4-Nitroaniline	.873091	2.78	ug/L	73605	85
8270WX	84	1,2-Diphenylhydrazine (as Azobenzene)	.9745878	3.248626	ug/L	34346	56
8270WX	86	4,6-Dinitro-2-methylphenol	.4713336	1.571112	ug/L	79533	47
8270WX	87	Dimethoate	.7694514	2.564838	ug/L	46314	42
8270WX	88	4-Aminobiphenyl	.67	2.23	ug/L	77581	5
8270WX	89	Diphenylamine/n-Nitrosodiphenylamine	1.43	4.77	ug/L	77579	55
8270WX	90	Pronamide	.7701194	2.567065	ug/L	39080	98
8270WX	91	Pentachloronitrobenzene	1.297181	4.323936	ug/L	81316	91
8270WX	93	Sulfotepp	.48020008	1.60067	ug/L	77871	101
8270WX	95	Cis-Diallate	.8229014	2.620705	ug/L	73540	112
8270WX	96	Phorate	.7579295	2.526432	ug/L	46313	96
8270WX	97	4-Bromophenyl-phenylether	.6273285	2.091095	ug/L	34636	19
8270WX	98	1,3,5-Trinitrobenzene	.5091168	1.697056	ug/L	73653	126
8270WX	99	Phenacetin	.9793584	3.264528	ug/L	73626	93
8270WX	100	trans-Diallate	1.527919	5.093062	ug/L	73540	125
8270WX	101	Hexachlorobenzene	.4494103	1.498034	ug/L	39700	62
8270WX	102	Pentachlorophenol	.7510706	2.503569	ug/L	39032	92
8270WX	103	Disulfoton	.6015917	2.005306	ug/L	81888	57
8270WX	104	Phenanthrene	.4097538	1.365846	ug/L	34461	94
8270WX	106	Anthracene	.4100871	1.366957	ug/L	34220	7
8270WX	107	Methyl Parathion	.6691773	2.230591	ug/L	39600	71
8270WX	108	Di-n-butylphthalate	.5770615	1.923538	ug/L	39110	30
8270WX	109	Ethyl Parathion	.8648028	2.882676	ug/L	39540	58
8270WX	110	4-Nitroquinoline-1-oxide	8.21	27.37	ug/L	73608	89
8270WX	111	Methapyrilene	1.023228	3.258688	ug/L	73589	70
8270WX	112	Isodrin	.4033251	1.644417	ug/L	39430	68

List

NAME	ESORT	PEAK	LOD	LOQ	UNITS	PCOD	RSOR
8270WX	113	Fluoranthene	.416354	1.368785	ug/L	34376	60
8270WX	115	Pyrene	.975	3.1	ug/L	34469	99
8270WX	116	Benzidine	.6093029	2.03101	ug/L	39120	10
8270WX	117	Aramite-A	1.2696	4.232	ug/L	73510	8
8270WX	118	Aramite-B	1.262345	4.207816	ug/L	73510	9
8270WX	119	p-(Dimethylamino)azobenzene	1.067397	3.557989	ug/L	73558	122
8270WX	121	Chlorobenzilate	1.054338	3.51446	ug/L	39460	23
8270WX	122	Butylbenzylphthalate	1.665082	5.550274	ug/L	34292	20
8270WX	123	3,3'-Dimethylbenzidine	.47	1.48	ug/L	73560	43
8270WX	124	2-Acetylaminofluorene	.7786698	2.595566	ug/L	73501	4
8270WX	125	3,3'-Dichlorobenzidine	.7676948	2.558983	ug/L	34631	38
8270WX	126	Benzo[a]anthracene	.4281667	1.427222	ug/L	34526	11
8270WX	127	Chrysene	1.158785	3.690399	ug/L	34320	28
8270WX	128	Bis(2-ethylhexyl)phthalate	1.130536	3.768455	ug/L	39100	110
8270WX	130	Di-n-octylphthalate	1.042319	3.474396	ug/L	34596	31
8270WX	131	Benzo[b]fluoranthene	.5635205	1.87	ug/L	34230	13
8270WX	132	7,12-Dimethylbenzo[a]anthracene	.4215279	1.405093	ug/L	73559	44
8270WX	133	Benzo[k]fluoranthene	.9044178	3.104726	ug/L	34242	15
8270WX	134	Benzo[a]pyrene	.4241883	1.413961	ug/L	34247	12
8270WX	135	Indeno[1,2,3-cd]pyrene	.543264	1.81088	ug/L	34403	67
8270WX	136	3-Methylchloranthrene	.4518533	1.506178	ug/L	73591	113
8270WX	137	Dibenzo[a,j]acridine	1.056896	3.522985	ug/L	q	33
8270WX	138	Dibenzo[a,h]anthracene	.5566482	1.8555494	ug/L	34556	32
8270WX	139	Benzo[g,h,i]perylene	.91	2.9	ug/L	34521	14

NAME	ESORT	PEAK	LOD	LOQ	UNITS	PCOD	RSOR
8270SX	1	Pyridine	288	960	ug/Kg	77045	97.5
8270SX	2	2-Picoline	19	64	ug/Kg	77088	97
8270SX	3	n-Nitrosomethylethylamine	34.1	113.7	ug/Kg	73613	116
8270SX	4	Ethyl Methanesulfonate	15.6	52	ug/Kg	73571	59
8270SX	5	n-Nitrosodimethylamine	30.6	102.1	ug/Kg	34438	78
8270SX	6	Methyl Methanesulfonate	28.4	94.7	ug/Kg	73595	72
8270SX	7	Aniline	22.8	76.2	ug/Kg	77089	6
8270SX	10	Phenol	42	140.3	ug/Kg	34694	95
8270SX	11	n-Nitrosodiethylamine	25.8	86	ug/Kg	78200	114
8270SX	12	Bis(2-chloroethyl)ether	25.9	86.4	ug/Kg	34273	18
8270SX	13	2-Chlorophenol	21.6	72	ug/Kg	34586	26
8270SX	14	1,3-Dichlorobenzene	30.1	100.4	ug/Kg	34566	37
8270SX	15	1,4-Dichlorobenzene	27.5	91.6	ug/Kg	34571	36
8270SX	16	1,2-Dichlorobenzene	26.9	89.7	ug/Kg	34536	35
8270SX	17	Benzyl Alcohol	30.8	102.8	ug/Kg	77147	17
8270SX	18	Bis(2-chloroisopropyl)ether	19.4	64.9	ug/Kg	73522	111
8270SX	19	2-Methylphenol	28.9	96.4	ug/Kg	77152	74
8270SX	20	n-Nitrosopyrrolidine	24.9	83	ug/Kg	78206	118
8270SX	21	n-Nitrosomorpholine	13.4	44.75	ug/Kg	73617	117
8270SX	22	0-Toluidine	19.3	64.5	ug/Kg	77142	121
8270SX	23	Hexachloroethane	23.5	78.4	ug/Kg	34396	65
8270SX	24	n-Nitroso-di-n-propylamine	14.7	49.1	ug/Kg	34428	76
8270SX	25	Acetophenone	17.7	59.1	ug/Kg	81553	3
8270SX	27	3 & 4-Methylphenol	40.7	135.9	ug/Kg	77146	75
8270SX	29	O,O,O-triethyl Phosphorothioate	24.7	82.4	ug/Kg	73652	124
8270SX	31	n-Nitrosopiperidine	17.1	57	ug/Kg	73619	79
8270SX	32	n-Nitrosodi-n-butylamine	18	60	ug/Kg	78207	77
8270SX	33	2,4-Dichlorophenol	23.9	79.7	ug/Kg	34601	40
8270SX	35	Nitrobenzene	27.2	90.9	ug/Kg	34447	86
8270SX	36	Isophorone	17	56.9	ug/Kg	34408	69
8270SX	37	2-Nitrophenol	20.3	67.8	ug/Kg	34591	87
8270SX	38	2,4-Dimethylphenol	37.3	124.5	ug/Kg	34606	45
8270SX	39	Bis(2-chloroethoxy)methane	18.3	61.2	ug/Kg	34278	109
8270SX	40	2,6-Dichlorophenol	18.5	61.8	ug/Kg	77541	39
8270SX	41	1,2,4-Trichlorobenzene	20.8	69.6	ug/Kg	34551	105
8270SX	42	Naphthalene	18.2	60.8	ug/Kg	34696	80
8270SX	43	4-Chloroaniline	27.5	91.9	ug/Kg	73529	22
8270SX	44	Hexachloropropene	20.1	67.1	ug/Kg	73576	66
8270SX	45	Hexachlorobutadiene	18.2	60.8	ug/Kg	34391	63
8270SX	46	a,a-Dimethylphenethylamine	300	1000	ug/Kg	73564	108
8270SX	47	p-Phenylenediamine	300	1000	ug/Kg	73628	123
8270SX	48	4-Chloro-3-methylphenol	18.9	63.2	ug/Kg	34452	21
8270SX	49	Cis-Isosafrole	17.1	57.2	ug/Kg	73582	29
8270SX	50	Safrole	17.7	59.2	ug/Kg	77545	100
8270SX	51	2-Methylnaphthalene	19	63.3	ug/Kg	77416	73
8270SX	53	Hexachlorocyclopentadiene	40.1	133.7	ug/Kg	34386	64
8270SX	54	1,2,4,5-Tetrachlorobenzene	15.1	50.6	ug/Kg	77734	102
8270SX	55	1-Chloronaphthalene	17.1	57	ug/Kg	38687	130
8270SX	56	4-Nitrophenol	40.1	133.8	ug/Kg	34646	88
8270SX	57	Pentachlorobenzene	14	46.9	ug/Kg	77793	90

NAME	ESORT	PEAK	LOD	LOQ	UNITS	PCOD	RSOR
8270SX	58	1-Naphthylamine	29	98	ug/Kg	73600	82
8270SX	59	2-Naphthylamine	29	98	ug/Kg	73601	81
8270SX	60	2,3,4,6-Tetrachlorophenol	53.8	179.4	ug/Kg	77770	103
8270SX	61	2,4,6-Trichlorophenol	17.3	57.8	ug/Kg	34621	106
8270SX	62	2,4,5-Trichlorophenol	18.6	62.1	ug/Kg	77687	107
8270SX	64	2-Chloronaphthalene	17.7	59.2	ug/Kg	34581	25
8270SX	65	2-Nitroaniline	19.6	65.5	ug/Kg	78142	84
8270SX	66	Acenaphthylene	16.4	54.9	ug/Kg	34200	2
8270SX	67	1,4-Naphthoquinone	55.01	183.3	ug/Kg	73599	119
8270SX	68	1,4-Dinitrobenzene	19.9	66.3	ug/kg	99149	131
8270SX	69	1,2-Dinitrobenzene	15.4	51.3	ug/kg	99148	132
8270SX	70	Dimethylphthalate	16.8	56.1	ug/Kg	34341	46
8270SX	71	1,3-Dinitrobenzene	30	102	ug/Kg	45622	48
8270SX	72	2,6-Dinitrotoluene	20	66.7	ug/Kg	34626	52
8270SX	73	Acenaphthene	15.1	50.4	ug/Kg	34205	1
8270SX	74	3-Nitroaniline	18.4	61.3	ug/Kg	78300	83
8270SX	75	2,4-Dinitrophenol	170	570	ug/Kg	34616	50
8270SX	76	Dibenzofuran	14.7	49.2	ug/Kg	81302	34
8270SX	77	2,4-Dinitrotoluene	22.3	74.6	ug/Kg	34611	51
8270SX	78	Fluorene	18.2	60.9	ug/Kg	34381	61
8270SX	79	4-Chlorophenyl-phenylether	13.9	46.5	ug/Kg	34641	27
8270SX	80	Diethylphthalate	15.4	51.5	ug/Kg	34336	41
8270SX	81	Thionazin	23.1	77	ug/Kg	73553	104
8270SX	82	5-nitro-o-toluidine	17.3	57.8	ug/Kg	73622	120
8270SX	83	4-Nitroaniline	14.1	47.2	ug/Kg	73605	85
8270SX	84	1,2-Diphenylhydrazine (as Azobenzene)	11.57	36.8	ug/kg	34346	56
8270SX	86	4,6-Dinitro-2-methylphenol	63.3	211	ug/Kg	79533	47
8270SX	87	Dimethoate	300	1000	ug/Kg	46314	42
8270SX	88	4-Aminobiphenyl	32	107	ug/Kg	77581	5
8270SX	90	Pronamide	19.1	63.8	ug/Kg	39080	98
8270SX	91	Pentachloronitrobenzene	17.4	58.11	ug/Kg	81316	91
8270SX	92	Diphenylamine/n-Nitrosodiphenylamine	36.4	121.58	ug/Kg	34433	115
8270SX	93	Sulfotepp	17.3	57.8	ug/kg	77871	133
8270SX	95	Cis-Diallate	20.9	69.78	ug/Kg	73540	112
8270SX	96	Phorate	18.3	61.28	ug/Kg	46313	96
8270SX	97	4-Bromophenyl-phenylether	16.4	54.8	ug/Kg	34636	19
8270SX	98	1,3,5-Trinitrobenzene	19.8	66	ug/Kg	73653	126
8270SX	99	Phenacetin	25.2	84	ug/Kg	73626	93
8270SX	100	trans-Diallate	45.5	151.8	ug/Kg	73540	125
8270SX	101	Hexachlorobenzene	15	50	ug/Kg	39700	62
8270SX	102	Pentachlorophenol	42.6	142.1	ug/Kg	39032	92
8270SX	103	Disulfoton	26.6	88.7	ug/Kg	81888	57
8270SX	104	Phenanthrene	15	50.2	ug/Kg	34461	94
8270SX	106	Anthracene	14	64	ug/Kg	34220	7
8270SX	107	Methyl Parathion	19	63.4	ug/Kg	39600	71
8270SX	108	Di-n-butylphthalate	20.1	67.1	ug/Kg	39110	30
8270SX	109	Ethyl Parathion	21.1	70.6	ug/Kg	39540	58
8270SX	110	4-Nitroquinoline-1-oxide	300	1000	ug/Kg	73608	89
8270SX	111	Methapyrilene	300	1000	ug/Kg	73589	70
8270SX	112	Isodrin	19	63	ug/Kg	39430	68

NAME	ESORT	PEAK	LOD	LOQ	UNITS	PCOD	RSOR
8270SX	113	Fluoranthene	15	47	ug/Kg	34376	60
8270SX	115	Pyrene	39	130	ug/Kg	34469	99
8270SX	116	Benzidine	14.34	45.64	ug/kg	39120	10
8270SX	117	Aramite-A	16.4	54.8	ug/Kg	73510	8
8270SX	118	Aramite-B	25.3	84.6	ug/Kg	73510	9
8270SX	119	p-(Dimethylamino)azobenzene	23.4	78	ug/Kg	73558	122
8270SX	121	Chlorobenzilate	18.6	62.3	ug/Kg	39460	23
8270SX	122	Butylbenzylphthalate	26.2	87.3	ug/Kg	34292	20
8270SX	123	3,3'-Dimethylbenzidine	43	143	ug/Kg	73560	43
8270SX	124	2-Acetylaminofluorene	17.6	58.6	ug/Kg	73501	4
8270SX	125	3,3'-Dichlorobenzidine	75	240	ug/Kg	34631	38
8270SX	126	Benzo[a]anthracene	18	57	ug/Kg	34526	11
8270SX	127	Chrysene	14.8	49.4	ug/Kg	34320	28
8270SX	128	Bis(2-ethylhexyl)phthalate	31	103.6	ug/Kg	39100	110
8270SX	130	Di-n-octylphthalate	24.4	81.4	ug/Kg	34596	31
8270SX	131	Benzo[b]fluoranthene	13.9	46.6	ug/Kg	34230	13
8270SX	132	7,12-Dimethylbenzo[a]anthracene	17.3	57.9	ug/Kg	73559	44
8270SX	133	Benzo[k]fluoranthene	22.1	73.66	ug/Kg	34242	15
8270SX	134	Benzo[a]pyrene	14.7	49	ug/Kg	34247	12
8270SX	135	Indeno[1,2,3-cd]pyrene	20.6	68.7	ug/Kg	34403	67
8270SX	136	3-Methylchloranthrene	15	50	ug/Kg	73591	113
8270SX	138	Dibenzo[a,h]anthracene	17.1	57	ug/Kg	34556	32
8270SX	139	Benzo[g,h,i]perylene	17.8	59.4	ug/Kg	34521	14

NORTHERN LAKE SERVICE, INC.

TITLE:

PREPARATION AND DETERMINATION OF CYANIDE USING MIDI-DISTILLATION & TECHNICON AUTO ANALYZER

SOP NUMBER: INO-METH-TECH-CYANIDE-1

EFFECTIVE DATE: NOV 1 2 2001

CONTROLLED COPY NUMBER: _____

Originated By: Kristina R. Hanney — NLS Associate — Date: 9/17/01

Approved By: Thomas K Reilly — Supervisor — Date: 09/14/01

Reviewed By: [signature] — QA Officer — Date: 9/18/2001

Authorized By: [signature] — Laboratory Manager — Date: 11.2.01

I. METHOD TITLE: Preparation and Determination of Cyanide Using Midi-Distillation & Technicon Auto Analyzer

II. METHOD SCOPE AND APPLICATION

A. The method covers the distillation of samples of drinking, ground, surface, and saline waters, domestic and industrial waste for the determination of the presence of cyanide. Once the samples are prepared, cyanide determination may be conducted by means of any appropriate manual, semi-automated or automated method.

B. NLS Test Code - 1900.

C. Detection Limit(s)

1. This method is appropriate for concentrations of cyanide ranging from 0.003 to 2.0 mg/L.

2. See current MDL study for detection limit.

III. REFERENCES

A. Greenberg, Arnold E., APHA, et al, Eds., Standard Methods for the Examination of Water and Wastewater,18th Edition. Washington D.C., American Public Health Association, 1992, p. 4-23.

B. METHOD FOR TOTAL CYANIDE ANALYSIS BY MIDI DISTILLATION (water and soils) 335.2 CLP-M (Semi-automated Spectrophotometric). USEPA Contract Laboratory Program Statement of Work for Inorganics Analysis, Doc. No. ILM01.0; D-91 D-98.

C. O'Dell, James W., Ed. 1993. Method 335.4: Determination of Total Cyanide by Semi-automated colorimetry. Methods for the Determination of Inorganic Substances in Environmental Samples; USEPA Office of Research and Development, EPA/600/R-93/100, 1993.

D. Andrews Glass Co. (July 15, 1994) Method For Preparing Drinking Water and Wastewater for Total Cyanide Determination with the Lab-Crest® "MIDI-DIST" ™ Midi Distillation System.

IV. METHOD SUMMARY

A. The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation process, and is absorbed in a sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by volumetric titration or colorimetrically.

B. Preliminary treatment of samples and appropriate colorimetric or titrimetric methods of analysis may be found in Standard Methods for the Examination of Water and Wastewater, 18th Edition, Section 4500-CN-B; in USEPA Contract Laboratory Program Statement of Work for Inorganics Analysis, Document Number ILM01.0; or in Methods for the Determination of Inorganic Substances in Environmental Samples, USEPA, August 1993, Method 335.4, Determination of Total Cyanide by Semi-automated Colorimetry.

C. The Lab-Crest® "Midi-Dist"™ Distillation System was developed to reduce the amount of sample and reagents required for analysis; significantly reduce the volume of waste generated and the risk of exposure to hazardous chemicals; maintain accuracy and sensitivity equal to the standard methods; simplify setup; and permit efficient preparation of multiple samples of water or wastewater for cyanide determination. The USEPA has assessed the Lab-Crest® "Midi-Dist"™ Distillation System, and considers it an acceptable minor modification to approved cyanide methodology when sample and

reagent rations are maintained. The method presented here will enable the analyst to easily adapt the standard method to the Midi-Dist.

V. INTERFERENCES

A. Some of the known interferences encountered with this method are aldehydes, nitrate-nitrite, oxidizing agents such as chlorine, thiocyanate, thiosulfate and sulfide. Some interferences are eliminated or reduced by the distillation.

B. Refer to 4500-CN-B., METHOD 335.2 CLP-M, or METHOD 335.4 for information on the preliminary treatment and preservation of samples and the treatment of interferences.

C. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other processing apparatus that bias analyte response.

VI. SAMPLING

A. Preservation and Storage

1. Preserve with NaOH to ph >12, keep cool at 4°C in plastic or glass.

B. Holding Times

1. 14 days.

VII. SAFETY

A. Safety for Midi-distillation unit:

1. Unit should be set up and operated in a chemical fume hood suitable for use with caustic and corrosive materials, hood face velocity should not be less than 100 CFM.

2. Wear hand and eye protection suitable for use with caustic and corrosive materials.

3. Hot surfaces during operation - Do not touch.

   a. Heat block operates at 125°C.
   b. Exterior case near heat block may exceed 80°C.

4. Do not move unit while hot, as sudden movement may result in bumping and sample loss.

5. Allow system to cool 20 minutes before removing glassware. Separation of "hot" glassware components may result in bumping, boil over, and spray of hot, corrosive materials.

6. Use excess gas trap in vacuum line to remove excess HCN vapor.

7. Avoid breathing vapors. May be harmful or fatal. Review all Material Safety Data Sheets for all materials used or generated in this operation.

8. If boil over occurs during operation, immediately wipe down unit with neutralizing solution, such as a mild sodium bicarbonate solution.

9. Unplug unit prior to cleaning exterior surfaces of unit (SHOCK HAZARD). Wipe with damp sponge or towel after each use, first with mild sodium bicarbonate or similar solution, followed by distilled water. Avoid excess solution on or near controls.

10. Water and vacuum service should be continued until unit is cool - boil over may otherwise result.

11. Use boiling chips in each distillation flask to prevent bumping.

B. General Lab Safety.

1. Use gloves while handling chemicals and reagents.

2. Must perform distillation under hood.

3. General lab safety rules apply.

VIII. EQUIPMENT AND MATERIALS

A. Apparatus

1. Lab-Crest® "Midi-Dist"™ Midi Distillation System with integrated heater, water and vacuum manifolds, timer, tubing and connectors, and reflux glassware designed for cyanide distillation.

2. Vacuum source and water source.

3. 500 mL excess cyanide trap

4. Appropriate equipment for colorimetric or titrimetric analysis.

IX. REAGENTS AND STANDARDS

A. Reagents for distillation.

1. Reagent water: Distilled or deionized, free of the analyte of interest.

2. Sodium Hydroxide Solution, 0.25 N: Dissolve 10 g NaOH in reagent water and dilute to 1L.

3. Magnesium Chloride Reagent, 51% (w/v): Dissolve 510 g $MgCl_2 \cdot 6H_2O$ in reagent water and dilute to 1L.

4. Sulfuric Acid, 18N: Slowly add 500 mLs of concentrated H_2SO_4 to 500 mL of reagent water. **Caution:** Mix under hood, extreme heat produced, mix slowly.

5. Lead Carbonate: $PbCO_3$, powdered.

6. Sulfamic Acid: NH_2SO_3H.

7. Lead acetate paper.

8. Acetate buffer, pH4: Bought prepared or dissolve 146 g anhydrous $NaC_2H_3O_2$, or 243 g $NaC_2H_3O_2 \cdot 3H_2O$ in 400 mLs of reagent water, add 480 g concentrated acetic acid and dilute to 1L with reagent water.

9. MBTH indicator solution: Bought prepared or dissolve 0.05 g 3-methyl,2-benzothiazolone hydrazone hydrochloride in 100 mL reagent water. Filter if turbid.

10. Ferric chloride oxidizing solution: Dissolve 1.6 g sulfamic acid and 1 g $FeCl_3 \cdot 6H_2O$ in 100 mLs reagent water.

11. Ethylenediamine solution, 3.5%: Dilute 3.5 mL anhydrous $NH_2CH_2CH_2NH_2$ to 100 mLs with reagent water.

12. Sodium Hydroxide Solution, 1N: Dissolve 2 g NaOH in reagent water and dilute to 1 L.

B. Reagents for analysis:

1. Sodium Phosphate Buffer: Dissolve 27.6 g sodium phosphate, monobasic crystal in 200 mLs of reagent water. Add 0.5 mL of Brij.-35. Shelf life 1 week.

2. Chloramine T: Dissolve 2.0 g chloramine-T (not Tri-hydrate) in 200 mLs of reagent water. Add 0.5 mL Brij.-35. Shelf life 1 week.

3. Pyridine Barbituric Acid: Bought prepared or place 15 g barbituric acid in a 250 mL volumetric flask and add just enough reagent water to wash sides of flask and wet barbituric acid. Add 75 mL pyridine and mix. Add 15 mL concentrate hydrochloric acid, mix, and cool to room temperature. Dilute to volume with reagent water and mix until barbituric acid is dissolved. Stable for 6 months if stored in amber bottle under refrigeration. Discard if precipitate develops.

C. Standards

1. 10 ppm working CN std: 1 ml of stock 1000 ppm std to 100 mLs with 0.05N NaOH.

2. Calibration standards:

a. 0.01 ppm calibration standard: 0.1 ml of 10 ppm WS to 100 mL with 0.05 N NaOH.

b. 0.05 ppm calibration standard: 0.5 ml of 10 ppm WS to 100 mL with 0.05 N NaOH.

c. 0.1 ppm calibration standard: 1.0 ml of 10 ppm WS to 100 mL with 0.05 N NaOH.

d. 0.2 ppm calibration standard: 2.0 ml of 10 ppm WS to 100 mL with 0.05 N NaOH.

X. PROCEDURE

A. Sample interference check.

1. Sulfide.

a. On a piece of Lead Acetate Paper premoistened with acetic acid buffer (pH 4.0) place one drop of sample. If paper turns black sulfide is present and must be corrected for. See method 4500-CN B. in Standard Methods for corrective action.

2. Aldehydes.

a. If the sample is alkaline, add 1 + 1 H_2SO_4 to 10 mL sample to adjust pH to less than 8. Place 1 drop of sample and 1 drop distilled water for a blank in separate cavities of a white spot plate. Add 1 drop MBTH solution and then 1 drop $FeCL_3$ oxidizing solution to each spot.

Allow 10 minutes for color development. The color change will be from a faint green-yellow to a deeper green with blue-green to blue at higher concentrations of aldehyde. The blank should remain yellow.

To minimize aldehyde interference, add 2 mL of 3.5% ethylenediamine solution/100 mL sample. This quantity overcomes the interference caused by up to 50 mg/L formaldehyde. When using a known addition in testing, 100% recovery of the CN- is not necessarily to be expected. Recovery depends on the aldehyde excess, time of contact, and sample temperature.

3. Other Interferences.

   a. See Method 4500-CN B. in Standard Method for other interferences and corrective actions.

B. Distillation Procedure:

1. Pour 50 mL of sample, or an aliquot diluted to 50 mL, into the reflux flask (to green ring). Add 2 or 3 boiling chips to prevent bumping.

2. Add 10 mL of 0.25 N NaOH and 40 mL of reagent water to absorber flask.

3. Connect the a) reflux flask, b) reflux impinger with air inlet and c) cold finger condenser, and the d) absorber flask and e) absorber impinger with medium porosity ROBU® frit, in the train as shown in Figure 1. Put the excess cyanide trap containing 250 mL 1.0 N NaOH in the vacuum line.

4. Start a slow stream of air entering the reflux flask by adjusting the vacuum needle valves until approximately 1 air bubble enters the reflux flask. This air rate will prevent a reverse flow of HCN through the air inlet. If this air rate does not prevent sample backup in the delivery tube, increase air flow rate to 2 or 3 air bubbles. Maintain air flow throughout the reaction.

5. Add 0.2 g of sulfamic acid (small scoop) through the air inlet tube, after the air rate is set. Let mix for 3 minutes.

6. Slowly pipette 5 mL 18 N sulfuric acid through the air inlet tube. Rinse tube with reagent water and let air mix contents for 3 minutes. Pipette 2.0 mL of magnesium chloride reagent through the air inlet and wash down with reagent water. A precipitate that may form redissolves on heating.

7. Switch on power to Lab Crest® "Midi-Dist"™ and set timer to one and one-quarter hours. Temperature is set at 125°C, and will permit rapid boiling. Do not flood condenser inlet or permit vapors to rise more than halfway into condenser. Adequate refluxing is indicated by a reflux rate of 40 to 50 drops/minute from the cold finger condenser bottom. Reflux for at least 1 hour. Timer will switch off heaters, but continue air flow for 15 minutes. Close off vacuum source and remove absorber flask when cool.

8. Determine cyanide concentration in the absorber solution by appropriate method, such as procedure in 4500-CN D, 3, or F; METHOD 335.2 CLP-M; or METHOD 335.4.

9. Distillation gives quantitative recovery of even refractory cyanides such as iron complexes. To obtain complete recovery of cobalticyanide, use ultraviolet radiation pretreatment. If incomplete recovery is suspected, distill again by refilling the absorber flask with a fresh charge of 0.25 N NaOH, and refluxing 1 hour more. The cyanide from the second reflux, if any, will indicate the completeness of the recovery.

10. As a quality control measure, periodically test apparatus, reagents, and other potential variables in the concentration range of interest. See METHOD 335.4 for greater detail about Quality Control, Calibration and Standardization and Safety procedures.

11. Place sample from absorber flask into 60 mL plastic bottle. Label and store in cooler until analysis.

XI. INSTRUMENT ANALYSIS

A. Equipment and Materials

1. Technicon Auto Analyzer II sampler.
2. Technicon Auto Analyzer II proportioning pump.
3. Technicon Auto Analyzer manifold.
4. Technicon Auto Analyzer S.C. Colorimeter.
5. Houston Instrument chart recorder.
6. ZEOS 386X Computer.
7. Software: Lt-notebook.
8. Okidata-printer.
9. General Lab glassware.

B. Procedure

1. Turn on instrument. Make sure waste jug is empty. Run deionized water for 5 - 10 minutes through lines.
2. Turn on colorimeter, set standard calibration at approximately zero - 1.00. Install 570 nm interference filters. Allow 30 minute warm-up time.
3. Make sure 60/hr, 4:1 cam is in sampler.
4. Turn on chart recorder. Settings should be 2 volts, chart speed 15 cm/hr. Check base line and full scale by running scale knob on colorimeter (recorder pen should go from 0 to full scale), then set to normal mode.
5. Place reagent lines into appropriate reagents. Lines are marked (PBA [Pyridine, Barbituric Acid], phosphate buffer, chloramine-T).
6. Allow base line to stabilize.
7. Fill sample cups with appropriate standards and blanks. Fill 2 cups with high standard to set standard calibration.
8. Fill 0.01, 0.05, 0.1, 0.2 mg/L standards and blank cups in duplicate. Set up samples or dilutions in remaining tray.

9. Turn on sampler by pushing start button. Let first 4 cups (with highest standard) come through and turn sampler off. Use these samples to set standard calibration peak height.

10. Turn instrument on and allow calibration standards to run. Turn chart recorder speed up to 50 cm/hr. As first peak starts (when sampler is on cup 4 - 5) press go button on computer to start notebook run.

11. At end of run turn off sampler, computer, charter recorder. Put reagent lines in deionized water to rinse.

C. Instrument Settings

1. Colorimeter - wave length 570 nm scale - normal.

2. Sampler - 60/hr, 4:1 cam.

3. Chart Recorder - 2 volts, 50 cm/hr.

4. Computer setup-Notebook.

a. Analysis - Qpro.

D. Calibration

1. Computer - Use Qpro to do calculations.

2. Hand calculate from chart recorder.

a. Use standards to create curve.

XII. CALCULATIONS

A. Number and measure height of peaks, either manually or using the data-station. Calculate all data by using one of three following methods:

1. Linear regression.

2. Plotting points on a curve using graph paper.

3. Quatro Pro (TECH.WKS) spreadsheet program.

B. Report 3 significant figures.

XIII. QUALITY CONTROL

A. Run verification standard at beginning and end of run.

B. Run check standard every 20 samples.

C. Run duplicate and spike every 20 samples, or at least one per day. To be set up before distillation.

1. If running spike, spike duplicate, use 1st spike to calculate spike recovery. Second spike value is for duplicate value only.

D. Set up blank and check standard (0.1 mg/L) with each distillation batch.

E. Distill verification standard for analysis.

XIV. RECORDS AND REPORTING DATA

A. Benchsheets and logbook entry

1. Enter all data on benchsheets.

2. Record any reagent and standard preparation in appropriate logbooks.

3. Record all distillation information and sample comments in distillation logbook.

4. Record blanks and check standard results in distillation logbook by appropriate distillation batch.

B. Units

1. mg/L.

C. Detection limits and Reporting limits.

1. See current MDL study.

D. Enter all data and QC into LIMS.

E. File all benchsheets, chart recordings, and computer printouts.

XV. CLEAN UP

A. Distillation cleanup.

1. Cleaning Glassware.

a. Clean glassware by rinsing with distilled water three times after each use. If necessary, use approved glassware cleaning agent with brush or automatic wash machine. If contamination persists, autoclave.

b. Clean absorber tube frit immediately after every use. Flush with distilled water forward and backwards three times. If frit becomes clogged, clean with concentrated hydrochloric acid for 20 minutes in an ultrasonic cleaner. (First place the Absorber Impinger(s) in a beaker and cover frit(s) with acid, then place beaker in ultrasonic cleaner.) If completely clogged, soak in Aqua Regia. Rinse with distilled water three times after any acid treatment.

2. Cleaning Heating Unit.

a. Unplug unit prior to cleaning.
IMPORTANT! Clean instrument after every use by wiping all exterior surfaces with a mild neutralizing agent such as sodium carbonate followed by distilled water. A damp sponge should suffice. Avoid excess solution on or near controls. In the event of a boil over or chemical spill, wipe unit immediately as best you can, followed by a thorough cleaning at the end of the run. The heat block holes should also be rinsed. Drain holes at the bottom of the block will allow spills and cleaning solutions to drain out to the bench below.
NOTE: This unit is acid resistant, not acid proof. Acidic vapors will coat exterior surfaces

even when no spills occur. Poor maintenance will result in a failure of the coating and will void warranty.

B. Auto Analzyer Cleanup.

1. Dump sample and throw sample cups away.
2. Disengage pump and loosen pump tube harness.
3. Turn off computer, printer, chart recorder and unplug heater.
4. Empty waste jug.
5. Wash any lab glassware used. Rinse with deionized water.

XVI. MAINTENANCE / TROUBLESHOOTING

A. Keep surfaces of auto analyzer and distillation unit clean.

B. Check all pump tubing for wear and replace all pump tubing when needed . Record in maintenance logbook.

C. Record all sample troubleshooting and corrective actions in appropriate logbook.

XVII. ATTACHMENTS

A. Worksheet for tray setup.

B. Diagram of distillation system.

Analyte		Analyst		Date		Cam		
Method		St. Cal.		File Name		Wavelength		
	Sample #	QC ID		Sample #	QC ID		Sample #	QC ID
1			1			1		
2			2			2		
3			3			3		
4			4			4		
5			5			5		
6			6			6		
7			7			7		
8			8			8		
9			9			9		
10			10			10		
11			11			11		
12			12			12		
13			13			13		
14			14			14		
15			15			15		
16			16			16		
17			17			17		
18			18			18		
19			19			19		
20			20			20		
21			21			21		
22			22			22		
23			23			23		
24			24			24		
25			25			25		
26			26			26		
27			27			27		
28			28			28		
29			29			29		
30			30			30		
31			31			31		
32			32			32		
33			33			33		
34			34			34		
35			35			35		
36			36			36		
37			37			37		
38			38			38		
39			39			39		
40			40			40		

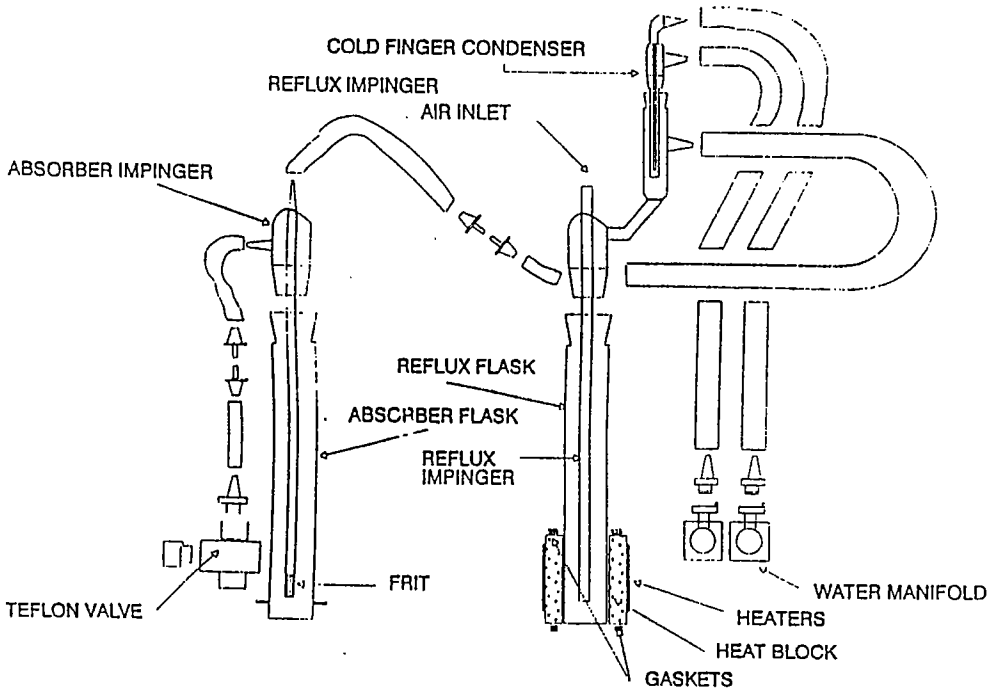


FIG 1. Lab-Crest "Midi-Dist"
Glassware and Tubing Assembly

NORTHERN LAKE SERVICE, INC.

TITLE:

COLORIMETRIC DETERMINATION OF HEXAVALENT CHROMIUM BY SPECTROPHOTOMETER

SOP NUMBER: INO-METH-SPEC-HEXCHROM-0

EFFECTIVE DATE: DEC 04 2000

CONTROLLED COPY NUMBER: _____

Originated By: Douglas M Jennings — NLS Associate — Date: 8-3-2000

Approved By: Thomas R Rhodes — Supervisor — Date: 8-7-00

Reviewed By: W Brooks Jr. — QA Officer — Date: 8-10-2000

Authorized By: [signature] — Laboratory Manager — Date: 12/4/2000

I. METHOD TITLE: Colorimetric Determination of Hexavalent Chromium by Spectrophotometer

II. METHOD SCOPE AND APPLICATION

A. NLS Test Codes

1. 1420

B. Detection Limit(s)

1. The current instrument reporting limit is 3.6 ug/L.

III. REFERENCES

A. EPA SW846-7196 "Test Methods for Evaluating Solid Waste" Third Edition.

IV. METHOD SUMMARY

A. This procedure measures only hexavalent chromium. Hexavalent chromium is determined colorimetrically by reaction with diphenylcarbazide in acid solution. A red-violet color of unknown composition is produced. The reaction is very sensitive; the molar absorptivity based on chromium being about 40,000 L/(g cm) at 540 nm. Total chromium can be determined by this method if a digestion is first performed by oxidation with potassium permanganate.

V. INTERFERENCES

A. The reaction with diphenylcarbazide is nearly specific for chromium.

B. Hexavalent molybdenum and mercury salts will react to form color with the diphenylcarbazide but the intensities are much lower than those for chromium. Concentrations as high as 200mg/L Mo or Hg can be handled.

C. Vanadium will interfere strongly but concentration up to ten times that of chromium will not cause trouble.

D. Permanganate interference is eliminated by reduction with the azide in the color reagent.

E. Interfering concentrations of these elements can be eliminated by extraction of the cupferrates of these metals into chloroform. If total chromium concentration is desired, this extraction is not recommended unless necessary. Residual cupferron and chloroform will complicate the oxidation with potassium permanganate.

VI. SAMPLING

A. Bottle Preparation - Glass or plastic.

B. Preparation – none.

C. Refrigeration - 4°C.

D. Holding Time - 24 hours.

VII. SAFETY

A. Use acceptable laboratory safety precautions and practices when handling reagents and samples.

VIII. EQUIPMENT AND MATERIALS

A. Glassware

1. 250 mL Erlenmeyer flasks.
2. 50 mL graduated cylinder.

B. Spectronic GENESYS 2™ spectrophotometer.

C. Eppendorf 1 mL pipette.

D. Filter apparatus with 0.45 um glass fiber filters.

IX. REAGENTS AND STANDARDS

A. Concentrated Sulfuric Acid.

B. Diphenylcarbazide Color Reagent

1. Dissolve 250 mg 1,5 diphenylcarbazide into 50 ml of acetone. Store the solution in an amber bottle and refrigerate at 4°C. Discard color reagent when the solution becomes discolored.

C. Chromium Stock Standard (50 mg/L).

1. Dissolve 141.4 mg Potassium Dichromate ($K_2Cr_2O_7$) in DI water and dilute to 1 liter.

D. Chromium Working Standard

1. Take 1 mL of 50 mg/L stock standard to 50 mL with DI water.

E. Calibration Standards

1. 20 ug/L – Dilute 1 mL working standard to 50 mL with DI water.
2. 40 ug/L – Dilute 2 mL working standard to 50 mL with DI water.
3. 60 ug/L – Dilute 3 mL working standard to 50 mL with DI water.

F. Laboratory Control Check Standard

1. Prepare a mid-range check standard using APG secondary standards. Prepare using directions provided by APG.

X. PROCEDURE

A. Calibration Curve

1. Set up calibration standards in Erlenmeyer flasks as instructed in section IX Reagents and Standards.
2. Include a blank (50 mL DI water) in the calibration curve.

B. Samples

1. Filter samples through a 0.45 um filter.
2. Pour 50 mL of sample filtrate into Erlenmeyer flask. If a dilution is required, dilute final volume to 50 mL with DI water.
3. For every batch of ten samples, a matrix spike and matrix spike duplicate must be analyzed.

    a. The sample should be spiked so that the spike concentration is at least 50% of the concentration of the high standard.

4. A lab control blank should be analyzed with the samples.

C. Color Formation

1. The pH of the calibrants and samples must be a 1.00 ± 0.3. To obtain this pH level, add 0.2 mLs of concentrated H_2SO_4 to the calibrants and samples.
2. Add 2.0 mLs of diphenylcarbazide solution to the calibrants and samples. Allow the calibrants and samples to set for 10 minutes, assuring complete color formation.

XI. INSTRUMENT ANALYSIS

A. Instrument Settings

1. The spectrophotometer should be pre-programmed at 540 nm.

B. Analysis of Samples on the Spectronic Genesys 2 Spectrophotometer

1. Turn power on (allow 30 minute warm up).
2. Pick **Standard Curve** application from **Main Menu**.
3. Press **# 1 Prepare Standard Curve.**
4. Press **Test Types.**

    a. Select **Hexi-Chrom.**
    b. Press **Exit.**

5. Calibration

    a. Zero instrument by inserting calibration blank and pressing **Auto Zero.**
    b. Press the number of the standard to measure or edit.
    c. Pour standard into cuvette and wipe off the cuvette with kimwipe.
    d. Make sure 8 cell holder is in position 1. Adjust with cell arrow keys.
    e. Place cuvette with sample into cuvette holder and close the door.
    f. Press **Measure Standard.**
    g. Press **Enter.**
    h. Empty and rinse out cuvette with blank.
    i. Repeat steps B through F until all of the standards are measured.
    j. Press the **print screen** button to print out raw calibration data.
    k. Press **Exit.**
    l. Press **View/Save Test** (calibration curve will be displayed.)
    m. Press **Save Test.**
    n. Press **Enter.**
    o. Calibration is complete, therefore, press **Exit** twice.

6. To determine the unknown concentrations press #2 Determination of Unknowns.

    a. Press **Sample ID.**

b. Press **1** and press **Exit**.
c. Make sure 8 cell holder is in position 1. Adjust with cell arrow keys.
d. Pour sample into cuvette and wipe off the cuvette with a kimwipe.
e. Place cuvette with sample into cuvette holder and close the door.
f. Press measure
g. Empty and rinse out cuvette with blank.
h. Press measure
i. Repeat steps D through H until all samples are read.
j. When samples are all read, exit all of the way out of the method.

7. Turn power switch off.

XII. CALCULATIONS

A. General Calculations

1. Multiply sample result with its appropriate dilution factor.

B. Significant figures

1. Report out to 3 significant figures.

XIII. QUALITY CONTROL

A. Matrix spike and matrix spike duplicate samples are analyzed every ten samples.

B. Matrix spike and matrix spike duplicate recovery data should be collected. When enough data points are gathered, a Students-T test will be performed to determine an acceptance range.

C. A laboratory blank sample is run with each batch of samples.

D. The laboratory blank analysis should be less than the reporting limit (3.6 ug/L).

E. A laboratory control check sample should be analyzed with each batch of samples.

F. The laboratory control check sample analysis results should be within the 99% confidence limits of the check standard.

NORTHERN LAKE SERVICE, INC.

TITLE:

DETERMINATION OF METALS BY TRACE ICP

SOP NUMBER: INO-METH-ICP/TRACE METALS-1

EFFECTIVE DATE: JUN 17 2002

CONTROLLED COPY NUMBER: _____

Originated By: _____ NLS Associate Date: 12 June 2002

Approved By: _____ Supervisor Date: 6-12-02

Reviewed By: _____ QA Officer Date: 6/12/2002

Authorized By: _____ Laboratory Manager Date: 6/14/02

I. METHOD TITLE: Determination of Metals by ICP and Trace ICP

II. METHOD SCOPE AND APPLICATION

A. Inductively coupled plasma-atomic emission spectroscopy (CP) determines trace elements, including metals, in solution. The method is applicable to all of the elements listed in Table 1. All matrices, including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis.

B. Elements for which Method 6010 is applicable are listed in Table 1. Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and model of spectrometer. Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

Table 1 - See Attachment A

III. REFERENCES

A. Winge, R.K.: Peterson, V.j.; Fassel, V.A. Inductively Coupled Plasma-Atomic Emission Spectroscopy: Prominent Lines (final report, March 1988-February 1978); EPA-600/4-79-017, Environmental Research Laboratory, Athens, GA, March 1989; Ames Laboratory: Ames IA.

B. Test Methods: Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater; U.S. Environmental Protection Agency. Office of Research and Development. Environmental Monitoring and Support Laboratory. ORD Publication Offices of Center for Environmental Research Information: Cincinnati, OH, 1982; EPA-600/4-82-057.

C. Patel, B.K.; Raab, G.A.; et al. Report on a Single Laboratory Evaluation of Inductively Coupled Optical Emission Method 6010; EPA Contract No. 68-03-3050, December 1984.

D. Sampling and Analysis Methods for Hazardous Waste Combustion; U.S. Environmental Protection Agency; Air and Energy Engineering Research Laboratory, Office of Research and Development: Research Triangle Park, NC, 1986; Prepared by Arthur D. Little, Inc.

E. Bowmand, P.W.J.M. Line Coincidence Tables for Inductively Coupled Plasma Atomic Emission Spectrometry, 2nd ed.; Pergamon: 1984.

F. Rohrbough, W.G.; et al. Reagent Chemicals, American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.

G. 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"' ASTM: Philadelphia, PA 1985; D1193-77.

H. Methods for Chemical Analysis of Water and Wastes. EPA March 1983, Method 200.7.

I. Standard Methods for the Examination of Water and Wastewater, 18th Ed. 1992.

J. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846 1986, (Method 6010)

IV. METHOD SUMMARY

A. Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods (e.g. Methods 3005-3050). When analyzing for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

B. Method 6010 describes the simultaneous, or sequential, multielemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by photomultiplier tubes. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate corrections made.

V. INTERFERENCES

A. Spectral interferences are caused by: (1) overlap of a spectral line from another element at the analytical or background measurement wavelengths; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuum or recombination phenomena; and (4) stray light from the line emission of high-concentration elements. Spectral overlap can be compensated for by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

Recommended wavelengths are listed in Table 1 and potential spectral interferences for the recommended wavelengths are given in Table 2. The data in Table 2 are intended as rudimentary guides for indicating potential interferences; for this purpose, linear relations between concentration and intensity for the analytes and the interferents can be assumed.

B. Element-specific interference is expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 2, 100 mg/L of Al would yield a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interference than those shown in Table 2. The interference effects must be evaluated for each individual instrument since the intensities will vary with operating conditions, power, viewing, height, argon flow rate, etc. The user should be aware of the possibility of interferences other than those specified in Table 2 and that analysts should be aware of these interferences when conducting analyses.

C. Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. Differences in solution volatility can also cause inaccuracies when organic solvents are involved. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, or diluting the sample. Changing the nebulizer and removing salt buildup at the tip of the torch sample injector can be used as an additional measure to control instrument drift. Also, it has been reported that better control of the argon flow rate improves instrument performance; this is accomplished with the use of mass flow controllers.

D. Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique. If observed, they can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition

procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

Table 2 - See Attachment B.

VI. SAMPLING

A. Bottle Preparation

B. Preservation - Samples must be preserved by addition of Nitric Acid to lower pH to <2.

C. Holding Times - Holding time of six months (180) days for preserved samples.

VII. SAFETY

A. Acids and metal salts in solution may be toxic/corrosive. Gloves, labcoats and safety glasses should be worn when handling these materials. Fume hoods are available and should be used when the situation requires. All personnel should be aware of the location of showers and eyewashes.

VIII. EQUIPMENT AND MATERIALS

A. Digestion/Extraction/Preparation Equipment

1. Inductively coupled argon plasma emission spectrometer:

   a. Computer-controlled emission spectrometer with background correction.
   b. Radio frequency generator compliant with FCC regulations.
   c. Argon gas supply - Welding grade or better.

2. Operating conditions:

   a. The analyst should follow the instructions provided by the instrument manufacturer. For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. All measurements must be within the instrument linear range where spectral interference correction factors are valid. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

3. Class A volumetric flasks.
4. Eppendorf mechanical pipettes.

IX. REAGENTS AND STANDARDS

A. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

1. Hydrochloric acid (conc), HCl.

2. Hydrochloric acid (1:10), HCl. Add 100 mL concentrated HCl to 400 mL water and dilute to 1 liter in an appropriate beaker.
3. Nitric acid (conc), HNO_3.
4. Nitric acid (1:1, HNO_3). Add 500 mL concentrated HNO_3 to 400 mL water and dilute to 1 liter in an appropriate beaker.

B. Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water will be interference free. Reagent water is defined as water that has been generated by any method which would achieve the performance specifications for ASTM Type II water.

C. Standard stock solutions are purchased.

D. Working standard preparation information is located in the standard preparation logbook for each instrument (Trace) (Atomscan 25).

X. PROCEDURE

A. Extraction/Digestion/Preparation

1. Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples which have been prefiltered and acidified will not need acid digestion as long as the samples and standards are matrix matched. Solubilization and digestion procedures are presented in Digestion SOP's.

XI. INSTRUMENT ANALYSIS

A. Instrument Settings

1. Set up the instrument with proper operating parameters as established.

B. Stabilization

1. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).

C. Calibration

1. Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions. Flush the system with the calibration blank between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.)

D. Sample/Standard Presentation to Instrument

1. Before beginning the sample run, reanalyze the highest mixed calibration standard as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 5% for 200.7 analysis (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition.
2. Flush the system with the calibration blank solution for at least 1 minute before the analysis of each sample. The blank must be ± MDL, or ± 3 x IDL for each element and the second source standard must be ± 10% of true value. See section 9.31 of Method 200.7 and section 8.6.1.3 of SW-846 6010.

   a. Interelement correction factor checks.

i. Trace ICP

(i.) A solution containing interferents and analytes should be run at the beginning (after calibration) and the end of the run. The measured concentrations should be within ± 20% of true value. See section 3.1.9 of SW-846 6010.

XII. CALCULATIONS

A. General Calculations

1. Sample above known linear range must be diluted. After dilution the resulting concentration must be multiplied by the dilution factor.

Example: 47 mg/L x 50 = 2350 mg/L

The sample was over range and was diluted 50 times. The MDL & LOQ must also be multiplied by the dilution factor (50).

B. Calculations for Solid Samples

$$C = \frac{r \times d \times v}{w \times s}$$

Where: c = concentration in mg/Kg.
r = sample result of digestate (ug/mL).
d = dilution factor.
v = final volume of digestate (mL).
w = weight of sample (g).
s = % solid of the sample (50% = 0.50).

Hardness (mg $CaCO_3$/L) = 2.497 [Ca, mg/L] + 4.118 [Mg, mg/L]

XIII. QUALITY CONTROL

A. Standards - Standard information can be found in standard preparation logbooks.

B. Sample QC

1. Spike recovery must be within NLS control limits.
   a) A spike with 100 µL will give a concentration of 100 ppb of Trace Metals and 1,000 ppb of Macro Metals in 10 mLs of sample.
2. Precision measurements must be within NLS control limits.
3. Failure of accuracy and precision measurements should refer to QC failure corrective action document.

XIV. RECORDS AND REPORTING DATA

A. Benchsheets function as run logs and data sheets.

B. Units - mg/L for Atomscan, ug/L for Trace ICP with some exceptions (Al, Ca, Fe, Mg, Na, K mg/L).

C. Detection limits are determined annually and can be located on a list of detection limits, in LIMS database, or on benchsheets.

D. Analytical data including benchsheets and instrument printout should be kept on file by date analyzed.

XV. CLEAN UP

A. Lab Work Area - Work areas should be cleansed after use.

B. Sample Disposal - Samples to be disposed of can be rinsed down the drain with copious amounts of cold water.

C. Containers can be rinsed and recycled.

XVI. MAINTENANCE/TROUBLESHOOTING

A. Preventive maintenance procedures

1. Daily

a. Peristaltic pump tubing should be replaced.
b. Waste carboy should be emptied as needed.
c. Sample uptake lines and nebulizer should be free of plugs.
d. Argon must have sufficient pressure and volume to complete the days analysis including warmup.
e. Instrument communications board and nebulizer pressure set switch should be reset.
f. Profile Trace ICP before beginning calibration.

B. Troubleshooting procedures - See operators manual

XVII. ATTACHMENTS

A. Table 1 Recommended Wavelengths and Estimated Instrumental Detection Limits.

B. Table 2 Analyte Concentration Equivalents Arising From Interference at the 100-mg/L level.

Table 1

Recommended Wavelengths and Estimated Instrumental Detection Limits

Detection Element	Trace ICP Wavelength[a](nm)	Detection Limit	AtomScan 25 Wavelength
Aluminum	308.215 (273.3)	*	**
Antimony	306.833	*	**
Arsenic	189.0	*	**
Barium	493.4	*	**
Beryllium	313.04	*	**
Boron	249.6	*	**
Cadmium	226.502	*	**
Calcium	317.933	*	**
Chromium	267.716	*	**
Cobalt	228.616	*	**
Copper	324.754	*	**
Iron	259.940 (271.4)	*	**
Lead	220.353	*	**
Lithium	N/A	*	670.8
Magnesium	279.079	*	**
Manganese	257.610	*	**
Molybdenum	N/A	*	202.0
Nickel	231.604	*	**
Potassium	766.491	*	**
Selenium	196.026	*	**
Silver	328.068	*	**
Sodium	588.995 (330.2)	*	**
Strontium		*	407.77
Thallium	190.864	*	**
Tin	N/A	*	189.9
Titanium	N/A	*	**
Vanadium	292.402	*	**
Zinc	213.856	*	**
*See Current List - Updated Yearly **See Current Method			

Table 2

Analyte Concentration Equivalents Arising From Interference at the 100-mg/L Level

Interferent[a,b] / Wavelength											
Analyte	(nm)	Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.3	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	213.856	--	0.05	--	0.005	--	--	--	--	0.02	--
Zinc	213.86	--	--	--	0.14	--	--	--	0.29	--	--

Interferent[a,b]
Wavelength ----------

[a]Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al - 1000 mg/L
Ca - 1000 mg/L
Cr - 200 mg/L
Cu - 200 mg/L
Fe - 1000 mg/L
Mg - 1000 mg/L
Mn - 200 mg/L
Ti - 200 mg/L
V - 200 mg/L

[b]The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferent figure.

NORTHERN LAKE SERVICE, INC.

TITLE:

DETERMINATION OF METALS BY ICP

SOP NUMBER: INO-METH-ICP-METALS-1

EFFECTIVE DATE: APR 19 2002

CONTROLLED COPY NUMBER: _____

Originated By: J. M. Cebe NLS Associate		Date: 4-10-02
Approved By: Thomas R. Rinke Supervisor		Date: 4-16-02
Reviewed By: W. J. Popek QA Officer		Date: 4/17/2002
Authorized By: Z. T. Mayer Laboratory Manager		Date: 4/18/2002

I. METHOD TITLE: Determination of Metals by ICP

II. METHOD SCOPE AND APPLICATION

A. Inductively coupled plasma-atomic emission spectroscopy (CP) determines trace elements, including metals, in solution. The method is applicable to all of the elements listed in Table 1. All matrices, including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis.

B. Elements for which Method 6010 is applicable are listed in Table 1. Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and model of spectrometer. Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

Table 1 - See Attachment A

III. REFERENCES

A. Winge, R.K.: Peterson, V.j.; Fassel, V.A. Inductively Coupled Plasma-Atomic Emission Spectroscopy: Prominent Lines (final report, March 1988-February 1978); EPA-600/4-79-017, Environmental Research Laboratory, Athens, GA, March 1989; Ames Laboratory: Ames IA.

B. Test Methods: Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater; U.S. Environmental Protection Agency. Office of Research and Development. Environmental Monitoring and Support Laboratory. ORD Publication Offices of Center for Environmental Research Information: Cincinnati, OH, 1982; EPA-600/4-82-057.

C. Patel, B.K.; Raab, G.A.; et al. Report on a Single Laboratory Evaluation of Inductively Coupled Optical Emission Method 6010; EPA Contract No. 68-03-3050, December 1984.

D. Sampling and Analysis Methods for Hazardous Waste Combustion; U.S. Environmental Protection Agency; Air and Energy Engineering Research Laboratory, Office of Research and Development: Research Triangle Park, NC, 1986; Prepared by Arthur D. Little, Inc.

E. Bowmand, P.W.J.M. Line Coincidence Tables for Inductively Coupled Plasma Atomic Emission Spectrometry, 2nd ed.; Pergamon: 1984.

F. Rohrbough, W.G.; et al. Reagent Chemicals, American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.

G. 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"' ASTM: Philadelphia, PA 1985; D1193-77.

H. Methods for Chemical Analysis of Water and Wastes. EPA March 1983, Method 200.7.

I. Standard Methods for the Examination of Water and Wastewater, 18th Ed. 1992.

J. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846 1986.

IV. METHOD SUMMARY

A. Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods (e.g. Methods 3005-3050). When analyzing for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

B. Method 6010 describes the simultaneous, or sequential, multielemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by photo multiplier tubes. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate corrections made.

V. INTERFERENCES

A. Spectral interferences are caused by: (1) overlap of a spectral line from another element at the analytical or background measurement wavelengths; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuum or recombination phenomena; and (4) stray light from the line emission of high-concentration elements. Spectral overlap can be compensated for by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

Recommended wavelengths are listed in Table 1 and potential spectral interferences for the recommended wavelengths are given in Table 2. The data in Table 2 are intended as rudimentary guides for indicating potential interferences; for this purpose, linear relations between concentration and intensity for the analytes and the interferents can be assumed.

B. Element-specific interference is expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 2, 100 mg/L of Al would yield a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interference than those shown in Table 2. The interference effects must be evaluated for each individual instrument since the intensities will vary with operating conditions, power, viewing, height, argon flow rate, etc. The user should be aware of the possibility of interferences other than those specified in Table 2 and that analysts should be aware of these interferences when conducting analyses.

C. Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. Differences in solution volatility can also cause inaccuracies when organic solvents are involved. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, or diluting the sample. Changing the nebulizer and removing salt buildup at the tip of the torch sample injector can be used as an additional measure to control instrument drift. Also, it has been reported that better control of the argon flow rate improves instrument performance; this is accomplished with the use of mass flow controllers.

D. Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique. If observed, they can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition

procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

Table 2 - See Attachment B.

VI. SAMPLING

A. Bottle Preparation – Pre-cleaned bottles are purchased and verified for cleanliness through analysis.

B. Preservation - Samples must be preserved by addition of Nitric Acid to lower pH to <2.

C. Holding Times - Holding time of six months (180) days for preserved samples.

VII. SAFETY

A. Acids and metal salts in solution may be toxic/corrosive. Gloves, lab coats and safety glasses should be worn when handling these materials. Fume hoods are available and should be used when the situation requires. All personnel should be aware of the location of showers and eyewashes.

VIII. EQUIPMENT AND MATERIALS

A. Digestion/Extraction/Preparation Equipment

1. Inductively coupled argon plasma emission spectrometer:

   a. Computer-controlled emission spectrometer with background correction.
   b. Radio frequency generator compliant with FCC regulations.
   c. Argon gas supply - Welding grade or better.

2. Operating conditions:

   a. The analyst should follow the instructions provided by the instrument manufacturer. For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. All measurements must be within the instrument linear range where spectral interference correction factors are valid. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

3. Class A volumetric flasks.
4. Eppendorf mechanical pipettes.

IX. REAGENTS AND STANDARDS

A. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

1. Hydrochloric acid (conc), HCl.

2. Hydrochloric acid (1:10), HCl. Add 100 mL concentrated HCl to 400 mL water and dilute to 1 liter.
3. Nitric acid (conc), HNO_3.
4. Nitric acid (1:1, HNO_3). Add 500 mL concentrated HNO_3 to 400 mL water and dilute to 1 liter.

B. Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water will be interference free. Reagent water is defined as water that has been generated by any method which would achieve the performance specifications for ASTM Type II water.

C. Standard stock solutions are purchased.

D. Working standard preparation information is located in the standard preparation logbook.

X. PROCEDURE

A. Extraction/Digestion/Preparation

1. Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples which have been prefiltered and acidified will not need acid digestion as long as the samples and standards are matrix matched. Solubilization and digestion procedures are presented in Digestion SOP's.

XI. INSTRUMENT ANALYSIS

A. Instrument Settings

1. See current instrument method for settings.

B. Stabilization

1. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).

C. Calibration

1. Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions. Flush the system with the calibration blank between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) See standards logbook for more detailed information.

D. Sample/Standard Presentation to Instrument

1. Before beginning the sample run, analyze a continuing calibration verification standard and a second source standard. Concentration values obtained should not deviate from the actual values by more than 10% (or the established control limits, whichever is lower). If they do, correct the problem, and recalibrate the instrument.
2. Flush the system with the calibration blank solution for at least 1 minute before the analysis of each sample. The blank must be ± MDL, or ± 3 x IDL for each element. See section 8.6.1.3 of SW-846 6010.
3. Analyze an interelement correction factor check standard. Choose the option that is appropriate for analyte selection and solution mix.

a. An interferent solution should be prepared and analyzed at the beginning of the run to verify automatic background correction. The analyte concentration (not interferent) displayed should be ± MDL, or ± 3 x IDL. If not calculate new IEC's.
b. A solution containing interferents and analytes should be run at the beginning of the run to verify automatic background correction. The measured concentrations should be within ± 20% of true value.
c. See standards logbook for more detailed information.

4. For samples requiring analysis by method 200.7, the following protocol is to be followed:

a. The continuing calibration verification standard must be within ± 5% of its true value upon initial reading. Subsequent analyses of this standard throughout the run must be within ± 10% of true value.
b. The continuing calibration blank will be < the analyte IDL, but > the lower 3 – sigma control limit of the calibration blank.
c. A Lab Fortified Blank (LFB) shall be analyzed, and found to be within ± 15% of the true value.

NOTE: If any of these requirements are not met, the analytical run does not qualify for method 200.7.

XII. CALCULATIONS

A. General Calculations

1. Sample above known linear range must be diluted. After dilution the resulting concentration must be multiplied by the dilution factor.

Example: 47 mg/L x 50 = 2350 mg/L

The sample was over range and was diluted 50 times. The MDL & LOQ must also be multiplied by the dilution factor (50).

B. Calculations for Solid Samples

1. $C = \frac{r \times d \times v}{w \times s}$

Where: c = concentration in mg/Kg.
r = sample result of digestate (ug/mL).
d = dilution factor.
v = final volume of digestate (mL).
w = weight of sample (g).
s = % solid of the sample (50% = 0.50).

Hardness (mg $CaCO_3$/L) = 2.497 [Ca, mg/L] + 4.118 [Mg, mg/L]

XIII. QUALITY CONTROL

A. Standards - Standard information can be found in standard preparation logbooks.

B. Sample QC

1. Spike recovery must be within NLS control limits.
2. Precision measurements must be within NLS control limits.

3. Failure of accuracy and precision measurements should refer to QC failure corrective action document.

XIV. RECORDS AND REPORTING DATA

A. Benchsheets function as run logs and data sheets.

B. Units - mg/L.

C. Detection limits are determined annually and can be located on a list of detection limits, in LIMS database, or on benchsheets.

D. Analytical data including benchsheets and instrument printout should be kept on file by date analyzed.

XV. CLEAN UP

A. Lab Work Area - Work areas should be cleansed after use.

B. Sample Disposal - Samples to be disposed of can be rinsed down the drain with copious amounts of cold water.

C. Containers can be rinsed and recycled.

XVI. MAINTENANCE/TROUBLESHOOTING

A. Preventive maintenance procedures

1. Daily

a. Peristaltic pump tubing should be replaced.
b. Waste carboy should be emptied as needed.
c. Sample uptake lines and nebulizer should be free of plugs.
d. Argon must have sufficient pressure and volume to complete the days analysis including warm up.
e. Instrument communications board and nebulizer pressure set switch should be reset.
f. Peak Search Atomscan25 before beginning calibration.

B. Troubleshooting procedures - See operators manual

XVII. ATTACHMENTS

A. Table 1 Recommended Wavelengths and Estimated Instrumental Detection Limits.

B. Table 2 Analyte Concentration Equivalents Arising From Interference at the 100-mg/L level.

Table 1

Recommended Wavelengths and Estimated Instrumental Detection Limits

Detection Element	Trace ICP Wavelength[a](nm)	Detection Limit	AtomScan 25 Wavelength
Aluminum	308.215 (273.3)	*	**
Antimony	306.833	*	**
Arsenic	189.0	*	**
Barium	493.4	*	**
Beryllium	313.04	*	**
Boron	249.6	*	**
Cadmium	226.502	*	**
Calcium	317.933	*	**
Chromium	267.716	*	**
Cobalt	228.616	*	**
Copper	324.754	*	**
Iron	259.940 (271.4)	*	**
Lead	220.353	*	**
Lithium	N/A	*	670.8
Magnesium	279.079	*	**
Manganese	257.610	*	**
Molybdenum	N/A	*	202.0
Nickel	231.604	*	**
Potassium	766.491	*	**
Selenium	196.026	*	**
Silver	328.068	*	**
Sodium	588.995 (330.2)	*	**
Strontium		*	407.77
Thallium	190.864	*	**
Tin	N/A	*	189.9
Titanium	N/A	*	**
Vanadium	292.402	*	**
Zinc	213.856	*	**
*See Current List - Updated Yearly **See Current Method			

Table 2

Analyte Concentration Equivalents Arising From Interference at the 100-mg/L Level

Analyte	Interferent[a,b] Wavelength (nm)	Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.3	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	213.856	--	0.05	--	0.005	--	--	--	--	0.02	--
Zinc	213.86	--	--	--	0.14	--	--	--	0.29	--	--

Interferent[a,b]
Wavelength ----------

[a]Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al - 1000 mg/L
Ca - 1000 mg/L
Cr - 200 mg/L
Cu - 200 mg/L
Fe - 1000 mg/L
Mg - 1000 mg/L
Mn - 200 mg/L
Ti - 200 mg/L
V - 200 mg/L

[b]The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferent figure.

NORTHERN LAKE SERVICE, INC.

TITLE:

Determination of Metals by Atomic Absorption - Flame or Furnace

SOP NUMBER: INO-METH-AA-FCE-0

EFFECTIVE DATE: JAN 2 1996

CONTROLLED COPY NUMBER: _____

Originated By: Douglas M Jennings NLS Associate Date: 12/20/95

Approved By: Thomas R Pielo Supervisor Date: 12/20/95

Reviewed By: W J Mack Jr. QA officer Date: 12/20/95

Authorized By: K. T. Lynn Laboratory Manager Date: 12/26/95

TEST: Determination of Metals by Atomic Absorption - Flame or Furnace.

REFERENCE: Methods for Chemical Analysis of Water and Wastes, EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

INSTRUMENTATION:

Varian AA-1475 Atomic Absorption Spectrophotometer.
Varian GTA-95 Graphite Tube Atomizer.
Perkin Elmer 4100ZL Zeeman Atomic Absorption Spectrophotometer.

SCOPE AND APPLICATION:

This method can be used for the determination of dissolved, suspended, or total elements in drinking water, surface water, waste water, and waste material. Dissolved metals are determined from filtered and acid preserved samples. Total metals are determined after appropriate digestion procedures have been performed. Drinking waters free of particulate matter may be analyzed directly. Domestic and industrial wastes require processing to solubilize suspended matter. Sludges, sediments and other solid samples can be analyzed after appropriate treatment.

Detection limits, sensitivity and optimum ranges will vary with various models of atomic absorption spectrophotometers. Table 1 and Table 2 contain the detection limits for the various metals analyzed on the Varian AA-1475 and the Perkin Elmer 4100ZL. Detection limits by direct aspiration may be extended through concentration of the sample and/or through solvent extraction techniques. Lower concentrations can also be determined through the furnace technique. When using the furnace technique, the analyst should be cautioned as to possible chemical reactions occurring at elevated temperatures which may interfere with the metals analysis. In order to provide valid data with furnace techniques, the analyst must examine each matrix for possible interference effects. If interference does occur in a particular matrix, the sample should be analyzed accordingly either through dilution, matrix modification, or the method of standard addition.

In samples where direct aspiration does not provide adequate sensitivity, the furnace technique may be used. Other specialized procedures are also available such as gaseous hydride method for arsenic and selenium, cold vapor method for mercury, and the chelation-extraction procedure for specific metals. Approved colorimetric methods for many elements are also available.

SUMMARY OF METHOD:

The following is a summary of the direct aspiration atomic absorption spectroscopy method and the furnace atomic absorption spectroscopy method. Lower detection limits can be determined with the furnace technique.

Direct Aspiration
In direct aspiration atomic absorption spectroscopy, a sample is aspirated into a flame. The heat of the flame causes the sample to atomize. A light beam from a hollow cathode lamp with a cathode made of the element of interest is directed through the flame into a monochromator. A detector in the monochromator measures the amount of light absorbed by the atomized sample. The amount of absorption depends on the presence of free unexcited ground state atoms in the flame. The light produced by the cathode lamp has a wavelength characteristic of the metal being analyzed. An increase in the element of interest in the flame causes more light to be absorbed. The detector monitors this absorption and a determination of the amount of element in a sample can be derived.

Furnace Technique
When using the furnace technique, a known amount of sample is placed in a graphite tube in the furnace. The graphite tube is then heated, causing the sample to evaporate, char, and finally atomize. Since a greater percentage of the atoms are atomized in the tube than in the flame, the detection of very low concentrations of elements is possible. The principle is very similiar to flame technique, except a furnace is used to atomize the sample instead of a flame. Light of a specific wavelength is directed through the vapor of the atomized sample. The intensity of the transmitted light decreases as the amount of ground state element increases. A monochromator isolates the wavelength of interest and transmits this to the photosensitive detector. The detector monitors the amount of absorption and a determination of the amount of element in the sample can be derived.

General Sample Characteristics
The atomic absorption method is generally limited to the analysis of metals in solution or solubilized through some form of processing. Wastewater and industrial effluents usually require some form of digestion because of the complexity and variability of the matrix. A digestion is required when the breakdown of organic material is necessary.

DEFINITIONS:

Optimum Concentration Range - A range below which scale expansion must be used and above which curve correction should be considered. This range will vary with the sensitivity of the instrument and the operating conditions.

Sensitivity - The concentration in milligrams of metal per liter that produces an absorption of 1%.

Detection Limit - Detection limits can be expressed as either instrument detection limits or method detection limits. Instrument detection limit is defined as the concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of a reagent blank signal at the same wavelength. Method detection limit is defined as the minimum concentration of a substance that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero and determined from analysis of a sample in a given matrix containing the analyte.

Dissolved Metals - Those constituents (metals) which will pass through a 0.45 um membrane filter.

Suspended Metals - Those constituents (metals) which are retained by a 0.45 um membrane filter.

Total Metals - The concentration of metals determined on an unfiltered sample following vigorous digestion if necessary, or the sum of the concentrations of metals in both the dissolved and suspended forms. Any samples containing visible particulates or solid material will require a digestion. Total metals, including Lead and Copper, in drinking water is performed on an unfiltered and undigested sample if the sample turbidity is less than 1 NTU.

Total Recoverable Metals - The concentration of metals in an unfiltered sample following treatment with hot dilute mineral acid.

SAFETY:
The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound should be treated as a potential health hazard. Exposure

to these chemicals must be reduced to the lowest possible level by whatever means available. A reference sheet for material handling is available for all chemicals used in this method. These sheets are available to all analysts.

INTERFERENCES IN FLAME ATOMIZATION:

The most common type of interference is chemical interference. This occurs when there is a lack of absorption of atoms bound in molecular combination in the flame. This can occur when the flame is not sufficiently hot enough to dissociate the molecules. The addition of lanthanum into the sample matrix will overcome the phosphate interference in the Magnesium, Calcium, and Barium determinations. The addition of Calcium will eliminate the Silica interference with Manganese. Chemical interferences may also be eliminated by separating the metal from the interfering material. While complexing agents are used to increase the sensitivity of the analysis, they may also be used to eliminate interferences.

INTERFERENCES IN FLAMELESS ATOMIZATION:

Although the use of flameless atomization greatly reduces oxide formation, it is still subject to chemical and matrix interferences. Gases produced in the furnace during analysis may have molecular absorption bands that overlap the analytical wavelength. The use of a background correction or an alternate wavelength should eliminate this problem.

Interferences from a smoke producing sample matrix can be reduced by extending the charring time at a higher temperature, or using an ashing cycle in the presence of air. Samples with high concentrations of organic material should be oxidized by a digestion procedure prior to analysis. Nitric acid is preferred for the digestion procedure. If the addition of other acids is required, use minimal amounts. This particularly applies to Hydrochloric acid.

EQUIPMENT:

The following is the equipment used to analyze water and waste samples for trace elements:

1. Varian AA-1475 Atomic Absorption Spectrophotometer.

2. Varian GTA-95 Graphite Tube Atomizer.

3. Perkin Elmer 4100ZL Zeeman Atomic Absorption Spectrometer.

3.5 Electrodeless Discharge Lamp (EDL) II Power Supply System.

4. Hollow Cathode Lamps (element specific).

4.5 Electrodeless Discharge Lamp (EDL) - Element specific.

5. Strip Chart Recorder, used primarily during Hg analysis.

6. Glassware.

REAGENTS AND STANDARDS:

The following is the required chemicals for atomic absorption analysis:

1. Distilled and deionized water.

2. Nitric Acid, concentrated.

3. Nitric Acid, (1:1) Add 500 mL of concentrated HNO_3 to 500 mL laboratory grade, distilled water.

4. Hydrochloric Acid, (1:1) Add 500 mL of concentrated HCL to 500 mL deionized, distilled water (for flame use only).

5. Stock standard metal solutions used for calibration and sample spiking.

6. Fuel and oxidant (for flame use only).

SAMPLE HANDLING AND PRESERVATION:

All laboratory glassware used in this procedure must be thoroughly washed with detergent and tap water; rinsed with (1:1) nitric acid; and final rinsed three times with distilled and deionized water. Chromic acid may be used to remove organic deposits from glassware, but extreme care must go into the final rinse in order to remove all traces of Chromium.

Before collection of the sample, a decision must be made as to what types of data is desired. For the determination of dissolved parameters, the sample must be filtered throught a 0.45 um membrane filter as soon as practical after collection and acidified to a pH <2.0 with (1:1) Nitric acid. For the determination of total or total recoverable parameters, the sample must be acidified to a pH <2.0 with (1:1) Nitric acid as soon as possible after the time of collection. For determinations of suspended parameters, a measured volume of nonpreserved sample must be filtered through a 0.45 um membrane filter as soon as possible after collection. The filter should then be transferred to a suitable container for storage. Drinking water samples containing suspended and setteable material should be prepared as a total recoverable parameter.

SAMPLE PREPARATION :

For the determination of dissolved parameters, the filtered, preserved sample may often be analyzed as received. Digestion is usually not required for a filter sample. The acid matrix and concentration of the samples and calibration standards must be the same. If a precipitate has formed upon acidification of the sample, the precipitate must be redissolved before analysis through the addition of more acid or by heat. If the precipitate can not be redissolved, the sample must be digested for total recoverable parameters.

Samples requiring total parameters for ground water, waste water, soils, and sludges must be digested prior to analysis. Follow the digestion procedures given on page DIG - 1.

PROCEDURE:

Instrument parameters specific to each element analyzed by atomic absorption are given in individual operating procedures in this manual. Refer to these procedures for instrument setups. The general start-up and analysis procedures for samples run on the Varian AA-1475 and the Perkin Elmer 4100ZL are given below.

I) Start-up procedure for Varian AA-1475 / GTA-95 Graphite Furnace.

1. Turn on exhaust fume hood (Hood is normally left on).

2. Turn argon carrier/purge gas on at tank requlator.

3. Turn printer on.

4. Turn power on to AA-1475 and GTA-95.

5. Select program to run on furnace.

   A. Pick 1 of 8 preset programs on instrument panel.
   B. Manually program element specific parameters.
   C. Element specific parameters begin on page AAM - 1.

6. Select proper Hollow Cathode Lamp and insert into turret.

7. Adjust lamp current to proper setting.

8. Allow lamp to warm up and stabilize for 15 - 45 minutes, depending upon the element of interest and choice of source lamp.

9. Check graphite tube appearance.

   A. Clean furnace head and quartz window if necessary.
   B. Insert pyrolytic platform or partition tube if replacement is necessary.

10. Select and optimize for element specific wavelength.

11. Optimize furnace workhead for maximum light throughput.

12. Optimize lamp for maximum light throughput using axial

    adjustment on lamp turret and recheck workhead alignment.

13. Change water in reagent blank/autosampler flush reservoir.

14. Purge air bubbles from autosampler syringe.

15. Insert appropriate modifiers, blanks, and standards into autosampler. Use the loading order list given below.

   A. Verify auto sampler positions and sample pickup, adjust if necessary.
   B. Verify sample injection into graphite tube.
      Adjust depth if necessary. Adjust autosampler if necessary.

16. When verification of autosampler is complete, reset the autosampler and restart the program with the appropriate samples inserted in the autosampler. Use the loading list given below.

17. Press run to initiate program.

18. Furnace will run a standard curve for the element of choice, followed by a verification standard and a blank.

19. If the results obtained for the calibration data and the from the analysis of the blank are in control, continue to analyze samples loaded according to loading list given below.

20. Follow QC requirements given at the end of this SOP.

II) Shutdown procedure for Varian AA-1475 and GTA-95.

1. Verify completion of last cycle.
2. Push STOP button on AA-1475.
3. Turn down lamp current if applicable.
4. Turn off GTA-95.
5. Turn off AA-1475.
6. Turn off Printer.
7. Turn off Argon gas at regulator.
8. Fume hood is normally left on.

III) Start-up procedure for Perkin Elmer 4100ZL.

1. Turn Argon gas on at tank regulator.
2. Turn printer on.
3. Turn computer on and start furnace software by executing AA-INSTRUMENT file.
4. Turn 4100ZL on.
5. Prepare fume extraction unit.

   A. Empty scrubber water container and refill with lab grad,e deionized water (if required).
   B. Remove scrubber filter paper and replace with new filter (if required).

5. Prepare distilled, deionized water reservoir and waste containers.

   A. Fill water reservoir.
   B. Drain waste reservoir and replace.

6. The EDL II source is now active.
7. Turn on the voltage supply and adjust to the proper voltage.
8. Allow 45 minutes for the EDL II's / HC's to warm up and stabilize.
9. Select the method to load into the furnace.
10. Pull down the <Align Lamps> menu and select the element to analyze. Select the <AGC/AIC> icon to get the intensity reading for the element. Align lamp for maximum intensity.
11. Select <Wavelength Scan> to verify the peak of interest.
12. Exit the <Align Lamps> window.

13. Prepare standards, blanks, modifiers, and verification standard and insert them in the proper locations in the autosampler. Use the loading list given below for sequence.

14. Select the <AS-70> window, and then select <Sampler Standby> and verify the sipper probe is entering the graphite tube properly. Use the X-Y Axis adjustments if needed.

15. Select <Flush Sampler> and remove impurities from the sipper.

16. Select <Calibrate> to initiate the run of the calibration curve. Visibly inspect and ensure proper sample deposition. Run the standard curve and observe peak shapes and correlation coefficients.

17. If the proper peak shapes are observed and the correlation coefficient is observed, calculate characteristic mass to verif the proper sensitivity.

18. Select the <AS-70> window and enter the positions required for the analysis.

19. Select <Run Samples> to initiate the run.

20. Use the loading list given below.

21. Verification standards recovery limits, QC limits, spikes recovery limits, and duplicate spike limits, are all preprogrammed in the method. They will be run automatically. If any deviations from the limits are encountered, the instrument automatically executes the corrective action required by the method.

22. Follow QC requirements given at the end of this SOP.

IV) Shutdown Procedure for Perkin Elmer 4100ZL.

1. Select the <AS-70> window, verify the current run has been completed, or select terminate run from the menu.

2. Turn down the voltage to the EDL II's.

3. Exit furnace software program.

4. Turn 4100ZL off.

5. Turn printer off.

6. Turn computer off

7. Turn off Argon gas supply at regulator.

IV) Sample Loading List Varian AA-1475 / GTA-95 Graphite Furnace.

1. The following is the loading list for the autosampler. These positions are relative to initial calibration and blank reagent samples.

Position Number	Sample Type
1	Check Standard
2	Blank
3	Sample 1
4	Sample 1 spiked
5	Sample 1 duplicate spike
6 - 10	Samples 2 through 6
11	Check Standard
12	Blank
13	Sample 7
14	Sample 7 spiked
15	Sample 7 duplicate spike
16 - 20	Samples 8 through 12

2. The same pattern holds true for positions 21 through 40. A blank and a check standard will be analyzed before every group of six samples.

V) Sample Loading List For Perkin-Elmer

1. The following is the loading list for the autosampler. These positions are relative to initial calibration and blank reagent samples.

Position Number	Sample Type
Check Std.	Check Standard
Blank	Blank
1a	Sample A
1b	Sample A plus spike
2a	Sample A duplicate
2b	Sample A duplicate plus spike
3-11	Samples
Check Std.	Check Standard
Blank	Blank
12a	Sample B
12b	Sample B plus spike
13a	Sample B duplicate
13b	Sample B duplicate plus spike
14-22	Samples
23a	Sample C
23b	Sample C plus spike
24a	Sample C duplicate
24b	Sample C duplicate plus spike
25-33	Samples

CALCULATIONS:

Direct determination of liquid samples can be derived from calibration curve linear regression analysis or from the direct readout of the instrument.If dilution of the sample was required:

ug/L metal in sample = A [(C + B) / C]

where A = ug/L of metal in the diluted aliquot from curve,
B = mL of deionized water used for dilution,
C = mL of sample aliquot.

If sample contains particulates:

ug/l metal in sample = A (V / C)

where A = ug/L of metal in the sample from curve,
V = final volume of the processed sample,
C = mL of sample processed.

For dry solid samples (reported as mg/kg dry weight basis):

mg metal / kg sample = (A x V) / D

where A = mg/L of metal in processed sample from curve,
V = final volume of the processed sample in mL,
D = weight of dry sample in grams.

For wet solid samples (reported as mg/kg dry weight basis):

mg metal / kg sample = (A x V) / (W x P)

where A = mg/L of metal in processed sample from curve,
V = final volume of the processed sample in mL,
W = weight of wet sample in grams,
P = percent solids as a decimal.

QUALITY CONTROL:

Instrument are standardized prior to each analytical run. All sample sets are bracketed by verification standards and blank check samples before and after the run. If verification standards and/or blanks are found to be out of control, possible sources of error will be checked and the samples will be reanalyzed using a new standard curve and all appropriate check standards and blanks. Spike recovery data must conform to established control limits that are established by the Northern Lake Service - Quality Control Database. If spike recovery data is found to be out of control, dilution of the sample and /or the method of standard additions will be employed to bring the sample into recovery limits or to qualify the data.

All quality control results from an analytical run must be entered into the Northern Lake Service Quality Control database. Control limits are recalculated every three to six months. Only those parameters with a minimum of thirty data points will be recalculated.

INSTRUMENT MAINTENANCE:

Procedures for general maintenance on the Varian AA-1475, the Varian GTA-95, and the Perkin Elmer 4100ZL are given in the owners manual for the instrument. Refer to these manuals for maintenance procedures.

INDIVIDUAL FURNACE ANALYSIS TECHNIQUES

1. ANTIMONY
2. ARSENIC
3. BERYLLIUM
4. CADMIUM
5. CHROMIUM
6. COPPER
8. LEAD
9. SELENIUM
10. SILVER
11. THALLIUM

TEST: Antimony by atomic absorption furnace technique.

REFERENCE: Methods for Chemical Analysis of Water and Wastes. EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE: 20 - 300 ug/l

DETECTION LIMIT: Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: The calibration standards should be matrix matched to the samples analyzed.

12.5 ug/L 25.0 ug/L 50.0 ug/L

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 60 seconds at 240 degrees Celsius.

2. Ashing time and temperature: 36 seconds at 1050 degrees Celsius.

3. Atomizing time and temperature: 10 seconds at 2000 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 217.6 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction is recommended.

2. Nitrogen may be used as a purge gas.

3. If Chloride presents a matrix problem, add an excess of 5 mg of ammonium nitrate to the furnace and ash using a ramp accessory or with incremental steps until the recommended ashing temperature is reached.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control quidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Arsenic by atomic absorption furnace.

REFERENCE:
Methods for Chemical Analysis of Water and Wastes. EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE:

5 - 100 ug/L

DETECTION LIMIT:

Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: The calibration standards should be matrix matched to the samples analyzed.

12.5 ug/L
25.0 ug/L
50.0 ug/L

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 60 seconds at 240 degrees Celsius.

2. Ashing time and temperature: 36 seconds at 100 degrees Celsius.

3. Atomizing time and temperature: 4 seconds at 2400 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 193.7 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame or Furnace".

NOTES:

1. Background correction may be required with high concentrations of dissolved solids.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control quidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Beryllium by atomic absorption furnace technique.

REFERENCE:

Methods for Chemical Analysis of Water and Wastes. EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE:

1 - 10 ug/L

DETECTION LIMIT:

Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: Calibration standards should be matrix matched to samples being analyzed.

1.0 ug/l 2.0 ug/l 3.0 ug/l

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 50 seconds at 250 degrees Celsius.

2. Ashing time and temperature: 35 seconds at 1000 degrees Celsius.

3. Atomizing time and temperature: 6 seconds at 2400 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 234.9 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction is recommended.

2. Nitrogen should not be used as a purge gas.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control quidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Cadmium by atomic absorption furnace.

REFERENCE:

Methods for Chemical Analysis of Water and Wastes. EPA March 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE:

0.5 - 3 ug/L

DETECTION LIMIT:

Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: Calibration standards should be matrix matched to samples being analyzed.

1.0 ug/L
2.0 ug/L
3.0 ug/L

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 45 seconds at 240 degrees Celsius.

2. Ashing time and temperature: 36 seconds at 700 degrees Celsius.

3. Atomizing time and temperature: 5 seconds at 1800 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 228.8 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction is recommended.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control quidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Chromium by atomic absorption furnace technique.

REFERENCE: Methods for Chemical Analysis of Water and Wastes. EPA March, 1983. Standard Methods For the Examination of Water and Wastewater 18th Edition Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE: 5 - 100 ug/L

DETECTION LIMIT: Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: Calibration standard should be matrix matched to samples being analyzed.

5.0 ug/L 10.0 ug/L 20.0 ug/L

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 40 seconds at 130 degrees Celsius.

2. Ashing time and temperature: 36 seconds at 1100 degrees Celsius.

3. Atomizing time and temperature: 4 seconds at 2500 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 357.9 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction may be required for samples high in dissolved solids.

2. Hydrogen peroxide added to the acidified solution will convert all Chromium to the trivalent state.

3. Nitrogen should not be used as a purge gas.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control quidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Copper by atomic absorption furnace.

REFERENCE:

Methods for Chemical Analysis of Water and Wastes, EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE:

1.0 - 60 ug/L

DETECTION LIMIT:

Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: Calibration standards should be matrix matched to samples being analyzed.

5.0 ug/l	10.0 ug/l	20.0 ug/l

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 30 seconds at 125 degrees Celsius.

2. Ashing time and temperature: 30 seconds at 900 degrees Celsius.

3. Atomizing time and temperature: 10 seconds at 2700 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 324.7 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction may be required with high concentrations of dissolved solids.

2. Nitrogen may be used as a purge gas.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control quidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Lead by atomic absorption furnace.

REFERENCE: Methods for Chemical Analysis of Water and Wastes. EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE: 1.0 - 60 ug/L

DETECTION LIMIT: Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: Calibration standards should be matrix matched to samples being analyzed.

5.0 ug/L 10.0 ug/L 20.0 ug/L

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 30 seconds at 125 degrees Celsius.

2. Ashing time and temperature: 30 seconds at 500 degrees Celsius.

3. Atomizing time and temperature: 10 seconds at 2700 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 283.3 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction is recommended.

2. Nitrogen may be used as a purge gas.

3. To suppress sulfate interference, lanthanum can be added to both samples and standards.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control quidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Selenium by atomic absorption furnace.

REFERENCE:

Methods for Chemical Analysis of Water and Wastes. EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE:

5 - 100 ug/L

DETECTION LIMIT:

Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: Calibration standards should be matrix matched to samples being analyzed.

12.5 ug/L
25.0 ug/L
50.0 ug/L

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 70 seconds at 250 degrees Celsius.

2. Ashing time and temperature: 36 seconds at 1100 degrees Celsius.

3. Atomizing time and temperature: 4 seconds at 2400 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 196.0 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction is recommended.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control quidelines given in the atomic absorption Standard Operationg Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Silver by atomic absorption furnace.

REFERENCE:

Methods for Chemical Analysis of Water and Wastes. EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE:

1.0 - 25 ug/L

DETECTION LIMIT:

Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: Calibration standards should be matrix matched to samples being analyzed.

2.5 ug/L 5.0 ug/L 10.0 ug/L

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 35 seconds at 240 degrees Celsius.

2. Ashing time and temperature: 36 seconds at 600 degrees Celsius.

3. Atomizing time and temperature: 4 seconds at 2000 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 328.1 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction may be required with high concentrations of dissolved solids.

2. Nitrogen may be used as a purge gas.

3. Avoid the use of halide acids.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control quidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

TEST: Thallium by atomic absorption furnace.

REFERENCE

Methods for Chemical Analysis of Water and Wastes. EPA March, 1983.
Standard Methods For the Examination of Water and Wastewater 18th Edition
Test Methods For Evaluating Solid Waste, Third Edition, Current Update.

OPTIMUM CONCENTRATION RANGE:

5 - 100 ug/L

DETECTION LIMIT:

Refer to current annual established Method Detection Limit listing.

STANDARD SOLUTIONS:

Prepare dilutions of the stock solution to provide the following calibration standards: Calibration standard should be matrix matched to samples being analyzed.

10.0 ug/L
20.0 ug/L
30.0 ug/L

INSTRUMENT PARAMETERS (General):

1. Drying time and temperature: 55 seconds at 240 degrees Celsius.

2. Ashing time and temperature: 36 seconds at 550 degrees Celsius.

3. Atomizing time and temperature: 5 seconds at 2200 degrees Celsius.

4. Purge gas atmosphere: Argon.

5. Wavelength: 276.8 nm.

6. Matrix modifiers as required.

ANALYSIS PROCEDURE:

Follow the analysis procedure given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NOTES:

1. Background correction is recommended.

2. Nitrogen may be used as a purge gas.

QUALITY CONTROL:

All duplicate and spike data generated during an analytical run must meet the control limit requirements established in the Northern Lake Service Quality Control Database. All QC data generated during an analytical run must be entered in this database. Follow quality control quidelines given in the atomic absorption Standard Operating Procedure "Metals by Atomic Absorption by Flame and Furnace".

NORTHERN LAKE SERVICE, INC.

TITLE:

DETERMINATION OF MERCURY BY ATOMIC ABSORPTION – COLD VAPOR TECHNIQUE

SOP NUMBER: INO-METH –AA-MERC/CV-2

EFFECTIVE DATE: JAN 3 0 2002

CONTROLLED COPY NUMBER: _____

Originated By: _____ Date: 1-17-02
NLS Associate

Approved By: Thomas K Ruede Date: 1-28-02
Supervisor

Reviewed By: _____ Date: 1/28/2002
QA Officer

Authorized By: _____ Date: 1/28/02
Laboratory Manager

I. METHOD TITLE: Determination of Mercury by Atomic Absorption - Cold Vapor Technique

II. METHOD SCOPE AND APPLICATION

A. Parameters

1. This method is applicable to groundwater, wastewater, soils, sediments, sludge, filters, and drinking waters. The cold vapor technique will measure mercuric ions only, and the digestion process employs potassium permanganate and potassium persulfate to oxidize the mercury compounds down to a measurable state.

B. NLS Test Codes / Descriptions

1. 2600, Hg dis.
2. 2620, Hg total.
3. 2622, Hg on extract.
4. 2655, Hg on TSP filters.

C. Detection Limit(s)

1. Aqueous
   a. See current MDL data for ug detection limit.
   b. Overall detection limit dependent on volume of sample used.
2. Solid/Sludge
   a. See current MDL data for ug detection limit.
   b. Overall detection limit dependent on amount of sample used.
3. Oil - not applicable.

III. REFERENCES

A. Methods for Chemical Analysis of Water and Wastes, EPA March, 1983. (Method 245.1)

B. Standard Methods for the Examination of Water and Wastewater – 20th Edition (Method 3112B)

IV. METHOD SUMMARY

A. Principles and Theories.

1. Mercury is measured based upon the absorption of radiation by mercury vapor at a wavelength of 253.7 nm. The mercury is reduced to mercuric ions and aerated from a closed system. The vapor passes through a flow cell positioned in the light path. Samples and standards are aspirated and absorbance is measured and recorded both manually and on a chart recorder.

V. INTERFERENCES

A. Matrix / Chemical Interferences

1. Samples high in organic material may not oxidize completely during the digestion phase. If this occurs, use a smaller sample size to ensure complete oxidation of samples. Interferences have been reported for waters containing sulfide, chloride, copper, and tellurium. Organic compounds, which have a broad band U.V. absorbance, (around 253.7 nm) are confirmed interferences. Volatile materials, which absorb at 253.7 nm, will cause a positive interference. Purge dead air space prior to addition of stannous chloride solution.

VI. SAMPLING

A. Bottle Preparation

1. Bottles used for collection of samples should be free from mercury contamination.

B. Preservation

1. Water samples should be preserved with NHO_3 to a pH of < 2.

2. Solid samples should be stored at 4°C.

C. Storage

1. Water samples can be stored either at room temperature or in a cooler.

2. Solid samples should be refrigerated.

D. Holding Times

1. Samples should be processed and analyzed within 28 days of collection.

VII. SAFETY

A. Special Precautions

1. Laboratory arrangement

a. Samples should be digested and cooled in a hood. Cooling samples may also be placed on a cart in front of a hood if the hood draw is adequate to pull vapors from cooling samples.

2. Chemicals

a. This process involves the use of strong acids, strong oxidizers, strong reducing agents, and toxic mercury vapors. All chemicals and samples should be handled with care. Refer to individual MSDSs for complete safety precautions.

3. Personal

a. Safety glasses, gloves, laboratory coat or apron, and a fume hood should be used.

VIII. EQUIPMENT AND MATERIALS

A. Digestion / Extraction / Preparation Equipment

1. Water bath capable of maintaining 95°C for 2 hours.

2. Repipette bottles for reagent and acid additions.

B. Glassware

1. Specifications

a. 300 mL BOD bottles and ground glass tops.

b. Disposable pipette tips and pipettor.

c. Aeration tube for BOD bottle purge.

2. Preparation

a. All glassware should be soap and water washed, tap water rinsed, HNO_3 rinsed, and triple-rinsed with distilled water.

IX. REAGENTS AND STANDARDS

A. Reagents

1. Sulfuric Acid (concentrated).

2. Nitric Acid (concentrated).

3. Stannous Chloride (100 g to 1,000 mL 10% Hydrochloric acid) as per S.M. 3112B.

4. Sodium Chloride - Hydroxlamine Sulfate Solution. (120 g of each to 1 L with distilled water)

5. Potassium Permanganate (50 g diluted to 1 L in deionized water).

6. Potassium Persulfate (50 g diluted to 1 L in deionized water).

7. Mercury Stock solution (100 ppm).

8. Mercury working solution (0.1 ppm Solution of Mercury Standard). Prepared fresh daily and acidify with HNO_3 to .15% acid.

9. Second source Mercury stock solution (100 ppm).

10. Mercury second source solution (0.1 ppm solution of Mercury standard). Prepare fresh daily and acidify with HNO_3 to 0.15% acid.

B. Reagent Purity Specifications

1. All reagents should be assayed by the chemical manufacturer for mercury and meet or exceed ACS specifications.

C. Standards Preparation Directions

1. Stock Hg std. at 100 mg/L concentration.

2. Working Hg std. at 0.10 mg/L concentration.

3. Second source stock Mercury standard at 100 ppm concentration.

4. Second source working standard at 0.1 ppm concentration.

   * Working standards should be prepared fresh daily.

Std	mL working std.	mL DI	Conc.
Blank	0.0	100	0.0 ug
0.05	0.5	100	0.05 ug
0.10	1.0	100	0.10 ug
0.20	2.0	100	0.20 ug
0.50	5.0	100	0.50 ug
1.00	10.0	100	1.00 ug

D. Storage Conditions

1. Stock mercury solution is good for one year from date opened.

X. PROCEDURE

A. Extraction / Digestion / Preparation

1. Sample preparation.

   a. Transfer 200 mL of sample or a weighed portion (approximately 0.2 grams dry weight) of solid/sludge to a 300 mL BOD bottle. Add 100 mL of distilled water to solid samples and an appropriate volume of distilled water to sludge samples to achieve a final volume of approximately 100 mLs.

   b. Add 5 mls working standard to the spike and spike duplicate to achieve a .50 ug concentration.

   c. Add 5 mL conc. H_2SO_4.

   d. Add 2.5 mL conc HNO_3.

   e. Add 15 mL Potassium Permanganate. (A Purple color must remain for 15 minutes).

   (Note 1: If the Purple color does not remain in a sample for 15 minutes, that sample must be reset using a smaller volume of sample until 15 mls of the Potassium Permanganate is adequate to establish a purple color that does remain for 15 minutes. Smaller sample volumes may be necessary for sewage or industrial wastewaters containing large amounts of chlorides.)

   (Note 2: Additional Potassium Permanganate may alternatively be added to samples that do not hold the purple color for 15 minutes, provided that the same amount is also added to all the standards and samples).

   f. Add 8 mL Potassium Persulfate $K_2S_2O_8$.

2. Sample Digestion.

   a. Mix sample thoroughly and heat for 2 hours in a water bath at 95°C.

   b. Allow samples to cool to room temperature before analysis.

XI. INSTRUMENT ANALYSIS

A. Instrument Settings

1. Varian AA-1475.

   a. Hg hollow cathode lamp, at approximately 3 mA.
   b. 253.7 nm wavelength, 0.5 slit width.
   c. 1.0 second read time.

2. Miscellaneous

   a. Absorption cell.
   b. Strip chart recorder.
   c. Aeration pump and associated tubing and glassware.
   d. Drying system to prevent water vapor condensation.

      i. Use heat lamp.
      ii. Use drying tubes with silica gel and/or magnesium perchlorate.

   e. Pipettors for addition of reagents.
   f. Distilled water wash bottle.
   g. Ground glass tops for 300 mL BOD bottles.

B. Stabilization

1. Start up Varian AA-1475 and allow instrument, hollow cathode lamp, and heat lamp to warm up for approximately 15 to 30 minutes.

2. Optimize wavelength, lamp, and flow cell for maximum light throughput.

3. Adjust baseline on chart recorder.

4. Step wise start up procedure.

   a. Turn on power switch.
   b. Install Hg hollow cathode lamp and set at 3 milliamps.
   c. Select a bandwidth of 0.5 nm.
   d. Select a wavelength of 253.7 nm.
   e. Install absorption cell on top of the burner head and set up aeration apparatus.
   f. Peak the wavelength, after optimizing the light path through the instrument.
   g. Install heat lamp over absorption cell.
   h. Warm up for 30 minutes.
   i. Turn pump on for an additional 10 minutes.
   j. Press auto zero.
   k. Turn graph on and begin standardization.

5. Step-wise shutdown procedure

   a. Verify completion of last sample or standard.
   b. Turn off graph.
   c. Turn off pump.
   d. Turn off power to AA and remove lamp.

C. Calibration/Analysis

1. To each BOD bottle, add 6 mL of sodium chloride – hydroxylamine sulfate solution.

   a. 120 g NaCl plus 120 g $(NH_2OH)_2 \cdot H_2SO_4$ diluted to 1 Liter.

2. Swirl solution to reduce all excess permanganate.

   a. If there is no excess of permanganate prior to the addition of the sodium chloride – hydroxylamine sulfate solution, the sample must be redigested using more permanganate or smaller sample size.

3. Attach the aeration apparatus to the BOD bottle and purge the dead airspace for approximately 15 - 30 seconds.
4. Add 5 mL Stannous Chloride solution to BOD bottle and immediately attach aeration apparatus. A water seal should be used on the ground glass joints.

   a. 100 g Stannous Chloride $(SnCl_2 \cdot 2H_2O)$ in 1 L 0.5 N H_2SO_4.

6. The absorbance of the sample will increase and reach a maximum within 30 seconds. This will be recorded manually and on the chart recorder. When the absorbance levels off and starts to fall, remove the aspiration from bottle and cap bottle with ground glass stopper. Place BOD bottle in hood and remove stopper to allow gasses to escape into hood.

7. Continue procedure for additional standards and samples until run is completed.

XII. CALCULATIONS

A. General Calculations

1. The standard curve for mercury determination is ug Hg vs. absorbance. Sample values are determined through a linear regression curve based on the various responses of blanks and calibration standards.

2. Sample values may also be calculated using peak height responses as measured from the graph of sample responses.

B. Significant figures

1. All data will be reported out using two significant figures.

C. Special Adjustments for Samples

1. All final values are based on the amount of sample used in the digestion step of the procedures. Various calculations are as follows:

   a. Liquids:

$$\frac{\text{ug Hg}}{\text{sample size (mL)}} \text{ X } \frac{1000 \text{ mL}}{1 \text{ L}} = \text{Hg in ug/L}$$

   b. Solids/Sludges on dry weight basis:

$$\frac{\text{ug Hg}}{\text{samples size (g) x \% solids as decimal}} = \text{Hg in mg/Kg DWB}$$

c. Solids/Sludge on wet weight basis:

$$\frac{\text{ug Hg}}{\text{sample size (g)}} = \text{Hg in mg/Kg WWB}$$

XIII. QUALITY CONTROL

A. Standards

1. Standard curve requires a minimum correction coefficient of 0.995.

B. Lab Control Standard (Verification Standard)

1. A verification standard from a separate source must be used.

2. Recovery limits are 90 - 110%.

C. Sample QC

1. Accuracy measurements (spike percent recovery):

a. Must comply with established limits set by NLS QA/QC, or qualified if matrix is suspect.

2. Precision measurements (duplicate - relative percent difference):

a. Must comply with established limits set by NLS QA/QC, or qualified if matrix is suspect.

D. Analytical Limitations

1. Sample Interferences

a. See Section V A.

2. Instrument Limitations

E. Method Validation and MDL study procedure

1. See established procedure.

XIV. RECORDS AND REPORTING DATA

A. Benchsheets and logbook entry - record all values on benchsheets.

B. Units / Significant figures - see current NLS policy.

C. Detection limits and Reporting limits - see current limits.

D. LIMS Entry - enter data into LIMS.

XV. CLEAN UP

A. Lab Work Area

1. Dismantle aeration equipment and place in proper storage area.

B. Sample Disposal

1. Samples are to be poured out under a hood.

C. Equipment / Glassware

1. All sample bottles should be washed with laboratory grade detergent, tap water rinsed, 1:1 HNO_3 rinsed, and triple rinsed with distilled water. Bottles should be drained thoroughly and capped with ground glass stoppers to prevent contamination.

XVI. MAINTENANCE / TROUBLESHOOTING

A. Preventive maintenance procedures and frequency

1. Hollow cathode lamps may need periodic replacement.

2. Quartz windows and flow cells should be inspected daily and cleaned when necessary.

NORTHERN LAKE SERVICE, INC.

TITLE:

DETERMINATION OF MERCURY BY COLD VAPOR GENERATION AND ATOMIC FLUORESCENCE DETECTION

SOP NUMBER: INO-METH-AF-MERCURY-2

EFFECTIVE DATE: OCT 15 2001

CONTROLLED COPY NUMBER: _____

Originated By: _____ Date: 7-17-2001
NLS Associate

Approved By: _____ Date: 8-15-01
Supervisor

Reviewed By: _____ Date: 8/16/2001
QA Officer

Authorized By: _____ Date: 10/15/01
Laboratory Manager

I. METHOD TITLE: Determination of Mercury by Cold Vapor Generation and Atomic Fluorescence Detection

II. METHOD SCOPE AND APPLICATION:

A. The mercury sample reacts with a solution of tin chloride ($SnCl_2$) to reduce it from a charged state to its vapor state. The vapor is purged from the aqueous phase using argon as the carrier gas and is removed from the liquid system with a high efficiency gas liquid separator. The mercury vapor is then passed over a membrane based drying tube to remove moisture and delivered to the atomic fluorescence detector.

B. The method determines total mercury.

C. The applicable range is 4 to 1000 ng Hg/L. The method reporting limit is 15 ng Hg/L for low-level mercury determinations and 0.05 ug Hg/L for standard mercury determinations.

III. REFERENCES

A. Determination of Mercury in Water by Semi-Automated Cold Vapor Atomic Fluorescence Spectrometry. EPA Method 245.7, Revision 1.1 June, 1994.

B. Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry. EPA Method 1631, EPA 821-R-96-001, January, 1996.

C. U.S. EPA, Methods for Determination of Metals in Environmental Samples, EPA –600/R-94/111, Revised May 1994, Method 245.2.

D. Ultra Clean Sample Handling, Nicholas S. Bloom, March/April edition of Environmental Lab 1995.

E. Guideline and Format for EMSL-Cincinnati Methods. EPA-600/8-83-020, August 1983.

F. Mercury Analysis in Soil/Sediment by Manual Cold Vapor Technique Method 245.5 CLP-M, Page no. D-56.

G. Monitoring Mercury Levels in the Environment: Application of a Fluorescence Approach for sub-ppb and ppt Levels. "American Environmental Laboratory" October, 1991, P.B. Stockwell; G. Vermeir; A. Grillo. Pp 14-20.

H. Lachat Instruments Quik Chem Method 10-138-37-1-A, Determination of Mercury by Cold Vapor Generation and Atomic Fluorescence Detection, January, 1996.

IV. METHOD SUMMARY

A. Mercury in the sample is reduced by a solution of tin chloride ($SnCl_2$) to reduce it from a charged state to an atomic vapor state. Mercury vapor is purged from the aqueous phase using argon as the carrier gas and is removed from the liquid stream with a high efficiency gas/liquid separator. The mercury vapor is then passed over a membrane type drying tube to remove moisture and delivered to the atomic fluorescence detector. The mercury vapor fluorescence at 254 nm is proportional to concentration.

V. INTERFERENCES

A. Mercury contamination of ambient air is a serious problem and must be considered for low level work. Rinse all glassware with a nitric acid rinse immediately before use to remove any residual mercury from the glass. Avoid using soft plastic containers for storage such as polyethylene or

polypropylene as the mercury will diffuse across these materials and result in a loss of sample or standard. Purge the stannous chloride reducing solution (reagent 1) for 15 – 20 minutes with argon to remove any mercury contamination from the stock reagents used in the preparation. This is done with the gas liquid separation rotameter set to 300.

VI. SAMPLING

A. Bottle Preparation – Samples should be collected in Teflon or glass bottles. All bottles must be thoroughly cleaned and rinsed per metals analysis cleaning procedures or purchased pre-cleaned. The volume collected must be sufficient to insure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.

B. Preservation – It is recommended that the sample be preserved with 1 mL of concentrated hydrochloric acid per 100 mL of sample for low-level mercury analyses. When standard mercury analyses are used along with other metals, 1.5 mL of nitric acid is added to a 500 mL sample bottle. Caution must be used as the addition of reagents may increase the risk of sample contamination and increase variability between sample replicates collected in the field.

C. Storage

1. Refrigeration – The mechanism for mercury absorption to the walls of the container, losses or absorption from the head space of the container, and the use of oxidants to prevent losses and stabilize the sample, have been reported. The storage of water samples refrigerated, acidified and preserved with oxidants have shown that mercury may be lost by absorption or gained by extraction from the atmosphere into the sample.

2. Ambient – It is recommended that the container lid be sealed tightly, placed in an isolation container, glove-bag or double container and refrigerated at 4° C.

D. Holding Times

1. Extraction/Digestion - The EPA suggests a holding time of 28 days or less

2. Analysis – For best sample results, samples are to be analyzed within 72 hours after collection. .

VII. SAFETY

A. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

B. A reference file of Material Safety Data sheets (MSDS) is available to all personnel involved in the chemical analysis.

C. The following chemicals have the potential to be highly toxic or hazardous; for detailed explanations consult the MSDS.

1. Mercury
2. Nitric acid (Trace-metal grade of low mercury content)
3. Hydrochloric acid (Trace-metal grade of low mercury content)
4. Sulfuric acid (Trace-metal grade of low mercury content)
5. Stannous chloride

VIII. EQUIPMENT AND MATERIALS

A. Digestion/Extraction/Preparation Equipment

1. Balance – analytical capable of accurately weighing to the nearest 0.0001 g.
2. Lachat Quik Chem Mercury Analyzer
3. Autosampler
4. Data system
5. Zero grade Ultra Pure Argon and Regulator

B. Glassware

1. Class A volumetric flasks and pipettes or plastic containers as required. Samples may be stored in Teflon or glass.

IX. REAGENTS AND STANDARDS

A. Reagent Purity Specifications

Use deionized water (10 megohm) for all solutions. All reagents should be prepared in nitric acid washed glass or equivalent containers to reduce potential contamination.

1. Reagent 1. Stannous Chloride Reducing Solution ($SnCl_2$). Anhydrous stannous chloride from Aldrich Chemical catalog #20,825-6 is recommended. If using an alternative source, please check that the solution is clear with no suspended white solid present before using.

   a. By Volume: In a 1 L volumetric flask add 20 g of solid anhydrous tin chloride ($SnCl_2$) to 200 mL of water, 80 mL of concentrated hydrochloric acid (HCl) and dilute to the mark with water. Purge with argon gas for 15 – 20 minutes before using to remove any mercury contamination. Prepare fresh daily.
   b. By Weight: In a 1 L container add 20 g of solid anhydrous tin chloride ($SnCl_2$) to 904 g water. Then add 94.4 concentrated hydrochloric acid (HCl). Purge with argon gas for 15 – 20 minutes before using to remove any mercury contamination. Prepare fresh daily.

2. Reagent 2. Blank Solution and Standards Diluent

   a. In a 1 L volumetric flask add approximately 700 mL water, then add 10 mL concentrated nitric acid (HNO_3). Dilute to the mark with deionized water after the solution has cooled. Prepare fresh daily. Prepare 3 liters of this reagent.

Required Reagents for the Bromate/Bromide Digestion

1. Reagent 3. Potassium Bromate ($KBrO_3$), (CASRN 7758-01-2)

   a. Volatilize trace metal impurities by heating in a muffle furnace at 250° C for a minimum of 8 hours. Place in a dessicator for cooling.

2. Reagent 4. Potassium Bromide (KBr), (CASRN 7758-02-3)

   a. Volatilize trace mercury impurities by heating in a muffle furnace at 250° C for a minimum of 8 hours. Place in a dessicator for cooling.

3. Reagent 5. Hydroxylamine Hydrochloride

   a. Dissolve 5 grams of hydroxylamine hydrochloride ($NH_2OH \cdot HCl$), in 100 mL of reagent water. Prepare fresh weekly.

4. Reagent 6. Bromate/Bromide Solution

   a. In a 500 mL volumetric flask dissolve 2.78 g potassium bromate and 11.90 g potassium bromide in 500 mL deionized water. Note: It is recommended that 0.1N potassium bromate/bromide solution, Cat. No. 35593, ALFA Chemicals, or equivalent be used. This solution may become contaminated with mercury from the laboratory atmosphere, therefore prepare only what can be used during a week. Long term storage is not recommended.

B. Standards Preparation Directions

To prepare the stock and working standards, the following container sizes will be required:

1. By Volume: four 100 mL and five 50 mL volumetric flasks.

It is recommended that the standards are prepared and stored in either glass or Teflon.

1. Standard 1. Stock Standard 1000 mg Hg/L.

   a. Purchase 1000 mg Hg/L stock standard (i.e. Spex CertiPrep or PE PureAtomic Spectrocopy Standard (N930-0174).

2. Stock Standard 2 in Reagent 2 (10 mg Hg/L)

   a. In a 100 mL volumetric flask add 1 mL of Standard 1 (1000 mg Hg/L). Dilute to the mark with Reagent 2 and invert to mix. Prepare fresh weekly.

3. Stock Standard 3 in Reagent 2 (100 µg Hg/L)

   a. In a 100 mL volumetric flask add 1 mL of Stock Standard 2 and dilute to the mark with Reagent 2 and invert to mix. Prepare fresh daily.

4. Stock Standard 4 in Reagent 2 (1.0 ug Hg/L)

   a. In a 100 mL volumetric flask add 1 mL stock standard 3 and dilute to the mark with Reagent 2 and invert to mix.

Low-level Mercury Standards

Working Standard (Prepare Daily) Concentration ug Hg/L	1.0	0.50	0.25	0.10	0.05	0.00

Volume (mL) of stock standard 3 diluted to 50 mL with reagent 2	0.5	0.25	0.125	--	--	--
Volume (mL) of stock standard 4 diluted to 50 mL reagent 2	--	--	--	5.0	2.5	--

Standard – level Mercury Standards

Working Standard (Prepare Daily) Concentration ug Hg/L	5.0	2.5	1.0	0.5	0.25	0.00

Volume (mL) of stock standard 3 diluted to 50 mL with reagent 2	2.5	1.25	0.5	0.25	0.125	--

X. PROCEDURE

A. Extraction/Digestion/Preparation

Potassium Bromate Bromide Digestion Procedure

1. Remove the individual sample from its isolation bag or double bags while in the instrument analysis room.

2. Prepare the conical vials by labeling the middle of the vial and caps. Do not interchange caps.

3. To the conical vial or the 50 mL volumetric flask used for digesting samples, add 5 mL (1:1) hydrochloric acid and 1 mL potassium bromate/potassium bromide solution (reagent 6).

4. For total recoverable mercury, transfer the sample (or aliquot of a sample) water to the vial or flask and fill to the 50 mL mark. Immediately cap the vial or flask after each filling. Check each vial and cap for a proper seal. Discard any vial and sample that leak and reprocess that sample.

5. For determination of dissolved mercury use either field filtered samples or filter through a 20 to 60 mL disposable syringe with an attached 0.45 µm filter. Remove the syringe plunger and pour the water sample into the syringe until it overflows. Replace the plunger and press the column of sample water through the filter, collecting the filtered water into the vial, filling to the 50 mL mark.

6. Allow samples to oxidize/digest for at least 2 hours before analysis. If the yellow color does not persist after 15 minutes, the sample may be diluted repeating sample preparation section, or additional potassium bromate bromide solution (1 mL) may be added if higher amounts of organic matter are present.

7. Stop the oxidation/digestion with the addition of 50 µL hydroxylamine hydrochloride (reagent 5) before storage or analysis.

XI. INSTRUMENT ANALYSIS

A. Instrument Settings

1. DATA SYSTEM PARAMETERS QUICKCHEM MERCURY ANALYZER

The system parameters listed below are approximate and will need to be optimized for individual applications.

Mercury System	Low Level	Standard Level
Sample throughput:	14 samples/h, 100 s/sample	27 samples/hour
Cycle Period:	250s	155 s
Purple pump tubes:	Stannous chloride	Stannous chloride
Orange pump tubes:	All other lines	All other lines
Gas Flow Rates:		
Carrier Flow:	300 mL/min	300 mL/min
Dryer Flow:	4 L/min	4 L/min
Sheath Flow (AF only):	300 mL/min	300 mL/min
Regulator gas:	30 – 35 lb/in2	30 – 35 lb/in2
Data System		
Gain Settings:		
Atomic Fluorescence:	range 100, gain 10	range 10, gain 10
Analyte Data:		
Concentration Units:	ng Hg/L	ug/L
Peak Base Width:	40 s	84
% Width Tolerance:	100	100
Threshold:	4000	100
Inject to Peak Start:	10 s	7 s
Chemistry:	Direct	Direct
Calibration Fit Type:	2nd Order Polynomial	2nd Order Polynomial
Calibration Rep.Handling:	Average	Average
Weighing Method:	No	No
Conc. Scaling:	None	None
Force Through Zero:	No	No
Sampler Timing:		
Min. Probe in Wash Period:	195 s	59 s
Probe in Sample Period:	55 s	55s
Valve Timing:		
Load Time:	0.0 s	0.00 s
Load Period:	45 s	35 s
Inject Period:	194 s	120 s

B. Calibration

1. Prepare reagent and standards as described.
2. Input data system parameters as indicated.
3. Pump deionized water through all reagent lines and check for leaks and smooth flow. Switch to reagents and allow the system to equilibrate until a stable baseline is achieved.

C. Sample/Standard Presentation to Instrument

1. Place samples and/or standards in the autosampler. Input the information required by the data system, such as concentration, replicates and QC scheme.
2. Calibrate the instrument by determining the standards. The data system will then associate the concentrations with the instrument responses for each standard.

D. System Notes

1. If sample concentrations are greater than the high standard, the digested sample should be diluted with diluent (Reagent 2). When the automated diluter is used, the diluent reservoir should contain reagent 2. Do not dilute digested samples or standards with deionized water.

2. The stannous chloride is available in different purity grades. The more pure the material the more difficult it is to get into solution. If the stannous chloride is not dissolving try adding 80 mL of concentrated hydrochloric acid to 20 grams of stannous chloride and let it boil for thirty minutes. Dilute the solution with 904 grams of water or dilute to one liter if preparing volumetrically. The solution should be clear at this point and should not require an argon purge. If the grade of stannous chloride being used is lower purity it may dissolve without difficulty resulting in a clear solution.

3. The argon used for low level work (less than 100 ng Hg/L) must be dry. If the argon contains moisture it may quench the signal in the AF detector resulting in decreased sensitivity.

XII. CALCULATIONS

A. General Calculations

1. Calibration is done by determining standards. The data system will then prepare a calibration curve by plotting responses versus standard concentration. Sample concentration is calculated from the regression equation.

2. Report only those values that are above the appropriate reporting levels of 10 or 50 ug Hg/L. Samples exceeding the highest standard should be diluted and reanalyzed.

B. Significant Figures

a. Report results in ng Hg/L. ng/L = ppt = parts per trillion.

XIII. QUALITY CONTROL

A. The minimum requirements of this program consist of an initial demonstration of laboratory capability, and the periodic analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated.

B. INITIAL DEMONSTRATION OF PERFORMANCE

1. The initial demonstration of performance is used to characterize instrument performance (determination of LCRs and analysis of QCS) and laboratory performance (determination of MDLs) prior to performing analyses by this method.

2. Linear Calibration Range (LCR) – The LCR must be determined initially and verified every 6 months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by +/- 10%, linearity must be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

3. Quality Control Sample (QCS) – When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. If the determined concentrations are not within ± 10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

4. Method Detection Limit (MDL) – MDLs must be established for all analytes, using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit. To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows: MDL = T x S, where t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.14 for seven replicates, t = 2.528 for twenty one replicates]. S = standard deviation of the replicate analyses.

   MDLs should be determined every six months, when a new operator begins work, or whenever there is a significant change in the background or instrument response.

C. ASSESING LABORATORY PERFORMANCE

1. Laboratory Reagent Blank (LRB) – The laboratory must analyze at least one LRB with each batch of samples. Data produced are used to assess contamination from the laboratory environment. Values that exceed the analysis Reporting Limit indicate laboratory or reagent contamination should be suspended and corrective actions must be taken before continuing the analysis.

2. Laboratory Fortified Blank (LFB) – The laboratory must analyze at least one LFB with each batch of samples. Calculate accuracy as percent recovery. If the recovery of any analyte falls outside the required control limits of 79 – 121% that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.

3. The laboratory must used LFB analyses data to assess laboratory performance against the required control limits of 79 – 121 %. When sufficient internal performance data become available (usually a minimum of 20 – 30 analyses), optional control limits can be developed from the percent mean recovery and the standard deviation (S) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

$$\text{UPPER CONTROL LIMIT} = \overline{X} + 3S$$

$$\text{LOWER CONTROL LIMIT} = \overline{X} - 3S$$

The optional control limits must be equal to or better than the required control limits of 79 - 121%. After each five to ten new recovery measurements, new control limits can be calculated using only the most recent 20 – 30 data points. Also, the standard deviation (S) data should be used to establish an on-going precision statement for the level of concentrations included in the LFB. These data are kept on file and are available for review.

4. Instruments Performance Check Solution (IPC) – For all determinations the laboratory must analyze the IPC (a mid-range check standard) and a calibration blank immediately following daily calibration, every tenth sample (or more frequently, if required) and at the end of the sample run. Analysis of the IPC solution and calibration blank immediately following calibration must verify that the instrument is within ± 10%. If the calibration cannot be verified within the specified limits, reanalyze the IPC solution. If the second analysis of the IPC solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift the instrument recalibrated. All samples following the last acceptable IPC solution must be reanalyzed. The analysis data of the calibration blank and IPC solution are kept on file with sample analyses data.

D. ASSESSING ANALYTE RECOVERY AND DATA QUALITY

1. Laboratory Fortified Sample Matrix (LFM) – The laboratory must add a known amount of analyte to a minimum of 10% routine samples. In each case the LFM aliquot must be a duplicate of the aliquot used for sample analysis. The analyte concentration must be high enough to be detected above the original sample and should not be less than four times the MDL. The added analyte concentration should be the same as that used in the laboratory fortified blank.

2. Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample, and compare these values to the designated LFM recovery range 90 – 110%. Percent recovery may be calculated using the following equation:

$$R = \frac{CS - C}{S} \text{X} 100$$

Where R = percent recovery, CS = fortified sample concentration, C = sample background concentration, S = concentration equivalent of analyte added to sample.

3. If the recovery of any analyte falls outside the designated LFM recovery range and the laboratory performance for that analyte is shown to be in control, the recovery problem encountered with the LFM is judged to be either matrix or solution related, not system related.

4. Where reference materials are available, they should be analyzed to provide additional performance data. The analysis of reference samples is performed at last quarterly for demonstrating the ability to perform the method acceptably.

XIV. RECORDS AND REPORTING DATA

A. Units/Significant Figures

1. Low-level - ng/L, three significant figures.
2. Standard level - ug/L; three significant figures.

B. Detection Limits and Reporting Limits

1. 15 ng/L (Low-level).
2. 0.050 ug/L (Standard level).

XV. CLEAN UP

A. Lab Work Area – The USEPA urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods, and bench operations, complying with the letter and spirit of any water discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel", available from the American Chemical Society.

B. Sample Disposal – The USEPA requires that laboratory waste management practice be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner.

C. Pollution Prevention

1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasible reduced at the source, the USEPA recommends recycling as the next best option.

2. The quantity of chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

3. For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is better: Laboratory Chemical Management for Waste Reduction." Available from the American Chemical Society's Department of Government Regulations and Science Policy, 115 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

XVI. ATTACHMENTS

A. Definitions

DEFINITIONS

1. CALIBRATION BLANK (CB) – A volume of reagent water in the same matrix as the calibration standards, but without the analyte.

2. CALIBRATION STANDARD (CAL) – A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

3. INSTRUMENT PERFORMANCE CHECK SOLUTION (IPC) – A solution of one or more method analytes used to evaluate the performance of the instrument system with respect to a defined set of criteria.

4. LABORATORY SPIKED BLANK (LSB) – An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LSB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

5. LABORATORY SPIKED SAMPLE MATRIX (LSM) – An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LSM is analyzed exactly like sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

6. LABORATORY REAGENT BLANK (LRB) – An aliquot of reagent water or other blank matrices that is digested exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. THE LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

7. LINEAR CALIBRATION RANGE (LCR) – The concentration range over which the instrument response is linear.

8. MATERIAL SAFETY DATA SHEET (MSDS) – Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

9. METHOD DETECTION LIMIT (MDL) – The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentrate is greater than zero.

10. PRACTICAL QUANTITATION LIMIT (PQL) – The lower level where measurements become quantitatively useful is the called the PQL. The PQL is defined as PQL = 10 x s, where s = the standard deviation of 21 replicates of a standard 2.5 – 5 times the MDL.

11. QUALITY CONTROL SAMPL (QCS) – A solution of method analytes of known concentrations that is used to spike an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared text materials.

12. STOCK STANDARD SOLUTION (SSS) – A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

13. USEPA – United States Environmental Protection Agency.

NORTHERN LAKE SERVICE, INC.

ATTACHMENT 11

LABORATORY QUALITY ASSURANCE SAMPLE HANDLING FORM EXAMPLES

- NLS Sample Collection and Chain of Custody Record Form
- NLS Order of Analysis Form / Preservation Guide / Documentation Guide
- NLS VOC's GRO/PVOC Sampling Instructions for Water
- NLS Sample Track (Internal QA Tracking Form)
- NLS Sample Tag (Label) Examples

SAMPLE COLLECTION AND CHAIN OF CUSTODY RECORD

NORTHERN LAKE SERVICE, INC.

Wisconsin Lab Cert. No. 721026460

Analytical Laboratory and Environmental Services

400 North Lake Avenue · Crandon, WI 54520-1298

Tel: (715) 478-2777 · Fax: (715) 478-3060

NO. 60033

CLIENT		
ADDRESS		
CITY	STATE	ZIP
PROJECT DESCRIPTION / NO.		QUOTATION NO.
CONTACT	PHONE	
PURCHASE ORDER NO.	FAX	

MATRIX:
SW = surface water
WW = waste water
GW = groundwater
TIS = tissue
AIR = air
DW = drinking water
SOIL = soil
SED = sediment
PROD = product
SL = sludge
OTHER

ANALYZE PER ORDER OF ANALYSIS

USE BOXES BELOW: Indicate Y or N if GW Sample is field filtered.
Indicate G or C if WW Sample is Grab or Composite.

ITEM NO.	NLS LAB. NO.	SAMPLE ID	COLLECTION DATE	COLLECTION TIME	MATRIX													COLLECTION REMARKS
1.																		
2.																		
3.																		
4.																		
5.																		
6.																		
7.																		
8.																		
9.																		
10.																		

ONE SAMPLE PER LINE

COLLECTED BY (signature)	CUSTODY SEAL NO. (IF ANY)	DATE/TIME
RELINQUISHED BY (signature)	RECEIVED BY (signature)	DATE/TIME
DISPATCHED BY (signature)	METHOD OF TRANSPORT	DATE/TIME

REPORT TO

RECEIVED AT NLS BY (signature)	DATE/TIME	CONDITION	TEMP.
COOLER #	REMARKS & OTHER INFORMATION		
	WDNR FACILITY NUMBER	E-MAIL ADDRESS	

INVOICE TO

PRESERVATIVE:
NP = no preservative
S = sulfuric acid
N = nitric acid
Z = zinc acetate
M = methanol
OH = sodium hydroxide
HA = hydrochloric & ascorbic acid
H = hydrochloric acid

IMPORTANT:

1. TO MEET REGULATORY REQUIREMENTS, THIS FORM MUST BE COMPLETED IN DETAIL AND INCLUDED IN THE SHIPPER CONTAINING THE SAMPLES DESCRIBED.
2. PLEASE USE ONE LINE PER SAMPLE, NOT PER BOTTLE.
3. RETURN THIS FORM WITH SAMPLES - CLIENT MAY KEEP PINK COPY.
4. PARTIES COLLECTING SAMPLE, LISTED AS REPORT TO AND LISTED AS INVOICE TO AGREE TO STANDARD TERMS & CONDITIONS ON REVERSE.

ORIGINAL COPY

Northern Lake Service, Inc
400 North Lake Avenue
Crandon, WI 54520-1298, (715) 478-2777 FAX: (715) 478-3060

Order of Analysis Form

(make additional copies if required)

Revised 6/19/02

SEND RESULTS TO:	CHAIN OF CUSTODY NUMBER:
	QUOTATION NUMBER:
	ELECTRONIC DATA SUBMITTAL: (circle) WDNR: TAD DMR ASCII Other:
SEND COPIES TO:	SEND INVOICE TO:

Indicate dissolved, total, or total recoverable analysis if applicable (circle appropriate analysis, or use D, T, TR)
Note "trace" for trace-level ICP analysis, and "F" for graphite furnace analysis.
Include detection limit requirements if they are available or applicable to your sample.
Include WDNR forms with samples if required.

Samples on Chain of Custody line #'s:_____ to be analyzed for the parameters checked below:

- ☐ Acidity
- ☐ Alkalinity, total
- ☐ Alkalinity, bicarb.
- ☐ Aluminum
- ☐ Antimony
- ☐ Arsenic
- ☐ Barium
- ☐ Beryllium
- ☐ BOD, 5 day (48 hr.)
- ☐ CBOD, 5 day (48 hr.)
- ☐ Boron
- ☐ Bromide
- ☐ Cadmium
- ☐ Calcium
- ☐ COD
- ☐ Chloride
- ☐ Chromium
- ☐ Chromium, hex. (24 hr.)
- ☐ Cobalt
- ☐ Coliform fecal(WW,24 hr.)
- ☐ Coliform total(DW,30 hr.)
- ☐ Color
- ☐ Conductivity
- ☐ Copper
- ☐ Cyanide, total
- ☐ Cyanide, amenable
- ☐ Fluoride
- ☐ Hardness
- ☐ Iron
- ☐ Iron bacteria
- ☐ Lead
- ☐ Lithium
- ☐ Magnesium
- ☐ Manganese
- ☐ Mercury
- ☐ Mercury, low level
- ☐ Molybdenum
- ☐ Nickel

Nitrogen Series

- ☐ Ammonia[NH_3]
- ☐ Nitrite[NO_2] (48 hr.)
- ☐ Nitrate[NO_3]
- ☐ Nitrate + Nitrite
- ☐ Kjeldahl (TKN)
- ☐ Total Nitrogen
- ☐ Organic Nitrogen

- ☐ Oil and Grease (HEM)
- ☐ pH
- ☐ Phenols
- ☐ Phosphorus, total
- ☐ Phos., tot. ortho (48 hr.)
- ☐ Phos., dis. ortho (48 hr.)
- ☐ Potassium
- ☐ Selenium
- ☐ Silica/Silicate as SiO2
- ☐ Silver
- ☐ Sodium
- ☐ Solids, total
- ☐ Solids, total dissolved
- ☐ Solids, total suspended
- ☐ Solids, total volatile
- ☐ Sulfate
- ☐ Sulfide
- ☐ **Surfactants**(MBAS, 48 hr.)*
- ☐ Thallium
- ☐ Tin
- ☐ Titanium
- ☐ TOC
- ☐ TOX
- ☐ Vanadium
- ☐ Zinc
- ☐ Municipal sludge list
- ☐ VOCs by 8260(8021/624)
- ☐ VOCs by 524.2(SDWA)
- ☐ BTEX by 8020
- ☐ PVOCs by 8020
- ☐ Naphthalene by 8020
- ☐ GRO+PVOC-WI mod.
- ☐ GRO-WI Mod.
- ☐ DRO-WI Mod.
- ☐ PAHs by 8310/610LC
- ☐ BNAs by 8270/625 (SVOC)
- ☐ Acid Extractables by 8270/625
- ☐ Base/Neutral Extractables by 8270/625
- ☐ Chlorinated Hydrocarbons by 8121/612
- ☐ Pesticides - Organochlorine by 8081/608
- ☐ PCBs by 8082/608
- ☐ Pesticides - Organophosphorus/ Nitrogen by 8141
- ☐ Acid Herbicides (Phenoxy) by 8151
- ☐ TCLP - Metals
- ☐ TCLP - Pesticides/Herbicides
- ☐ TCLP - BNA's
- ☐ TCLP - VOC's
- ☐ SPLP - Metals
- ☐ SPLP -
- ☐ ASTM - Metals
- ☐ ASTM - _____
- ☐ _____
- ☐ _____
- ☐ _____

Fecal coliform analysis for sludge samples arriving in quantities of 7 require lab notification.

* **Surfactants require laboratory notification prior to sample receipt. A 2x surcharge is applied to samples received without notification.**

Special instructions: _____

See reverse for example chain of custody record keeping

Sample Bottle and Preservation Guide

Revised 6/18/02

Inorganic analyses, water

Plastic, Non-preserved
Acidity
Alkalinity
Bromide
Choride
Fluoride
Nitrite (48 hrs.)
Sulfate
BOD
CBOD
Chromium, hex.
Color
Conductivity
pH
Orthophosphate
Solids, total
Solids, total suspended
Solids, total volatile
Surfactants, (MBAS 48 hr.)

Glass, H_2SO_4
(2 Bottles per sample)
Phenols

Plastic, NaOH
Cyanide, total
Cyanide, ammenable

Plastic, sterile
Coliform, total (30 hr.)
Coliform, fecal (24 hr.)
Iron Bacteria

Plastic, HNO_3
Metals analysis
Mercury
Hardness

Glass, HCl bubble wrapped and bagged
Mercury, low level 15 ng/L

Plastic, H_2SO_4
Nitrate, uncorrected
Nitrate + Nitrite
Kjeldahl (TKN)
Ammonia
Total Nitrogen
Organic Nitrogen
Total Phosphorus
COD

Plastic, Zn Acetate + NaOH
Sulfide

Organic Analyses, water

Glass, Clear, HCl, 1000 mL
(2 Bottles per sample)
Oil and Grease, hexane

Glass, Amber, 300mL, H_2SO_4
TOX

Glass, Amber, HCl, 1000mL
(2 Bottles per sample)
DRO-WI mod

VOC vials, Clear, HCl, 40 mL
(2 Bottles per sample)
VOCs by 8260(8021/624)
VOCs by 524.2(SDWA)
PVOCs by 8020
GRO WI mod
BTEX by 8020
Naphthalene

Glass, Amber, 1000mL
(2 Bottles per sample)
TCDD/TCDF

Plastic, H_2SO_4, 125 mL
TOC

Glass, Amber, Non-preserved
(2 Bottles/sample except mult. param)
Acid extractables 8270/625
Base/Neutrals 8270/625
BNA's 8270/625
PAH's 8310/610LC
Chorinated Hydrocarbons 8121/6012
Pesticides - organochlorine 8081/608
PCB's 8082/608
Pesticides - organophosphorus 8141
Phenols by GC 8040/604
Acid herbicides (phenoxy) 8151

TCLP Parameters
- Metals (250 grams)
- Pest/Herb — Glass
- BNA's — Amber
- VOC's — 1000 mL

SPLP
- Metals (250 grams)
- Other________

ASTM
- Metals (250 grams)
- Other________

Inorganic analyses, soil/solid

Plastic, non-preserved sludge jar
All analytes
TCLP - 250 grams

Plastic, non-preserved, 60 mL
Percent solids for DWB calculations
Most metals combinations

Organic analyses, soil/solid

Glass, Amber, 60 mL
(Bottle count)
DRO (2)
PAH (1)
PCB (1)
Pesticides (1)
BNA (1)

Plastic, non-preserved
TOX

Glass, Amber, 60 mL
(2 Bottles per sample)
PVOC, VOC, GRO — (MeOH preservative is packaged seperately)

Example of appropriate chain of custody documentation

SAMPLE COLLECTION AND CHAIN OF CUSTODY RECORD
Wisconsin Lab Cert. No. 721026460

NORTHERN LAKE SERVICE, INC.
Analytical Laboratory and Environmental Services
400 North Lake Avenue • Crandon, WI 54520-1298
Tel: (715) 478-2777 • Fax: (715) 478-3060

NO. 56000

CLIENT: ANYTOWN WASTEWATER UTILITY
ADDRESS: 100 1ST STREET
CITY: ANYTOWN — STATE: WI — ZIP: 55555
PROJECT DESCRIPTION / NO.: QUARTERLY WASTEWATER — QUOTATION NO.: 12345
CONTACT: JOHN DOE — PHONE: 555-555-5555
PURCHASE ORDER NO.: 54321 — FAX: 555-555-5505

MATRIX: SW, WW, GW, TS, AIR, DW, SOIL, SED, PROD, SL, OTHER

USE BOXES BELOW: Indicate Y or N if GW Sample is field filtered. Indicate G or C if WW Sample is Grab or Composite.

ITEM NO.	LAB NO.	SAMPLE ID	COLLECTION DATE	COLLECTION TIME	MATRIX	ANALYZE PER ORDER OF ANALYSIS	PHOSPHORUS, TOTAL	AMMONIA, TOTAL	N+N, TOTAL	CHLORIDE, DISSOLVED	ALKALINITY, DISS.	PH, LAB	VOC-524.2	ALKALINITY, TOT.	TSS			COLLECTION REMARKS
							G	G	G	Y	Y	Y	G	G	G			
1.		EFFLUENT	1/1/01	09:05	WW		X	X	X									EFFLUENT COLLECTED
2.		INFLUENT	1/1/01	09:15	WW		X	X	X									BEFORE INFLUENT
3.		(WASTEWATER EXAMPLE)																
4.																		
5.		MONITORING WELL 1	1/1/01	10:40	GW					X	X	X						HEAVY RAIN DURING
6.		MONITORING WELL 2	1/1/01	11:22	GW					X	X	X						MW2 COLLECTION
7.		(GROUNDWATER EXAMPLE)																
8.																		
9.		PRIVATE WELL	1/1/01	13:05	DW								X	X	X			
10.		(DRINKING WATER EXAMPLE)																

COLLECTED BY (signature): John Doe — CUSTODY SEAL NO. (IF ANY) — DATE/TIME
RELINQUISHED BY (signature) — RECEIVED BY (signature) — DATE/TIME
DISPATCHED BY (signature): John Doe — METHOD OF TRANSPORT: UPS, FED EX, ETC. — DATE/TIME: 1-1-01 / 15:30
RECEIVED AT NLS BY (signature) — DATE/TIME — CONDITION — TEMP.
COOLER # — REMARKS & OTHER INFORMATION
WDNR FACILITY NUMBER: 721026460 — E-MAIL ADDRESS

REPORT TO: ATTN: JOHN DOE, ANYTOWN WASTEWATER UTILITY
INVOICE TO: ANYTOWN WASTEWATER UTILITY

PRESERVATIVE: NP = no preservative; S = sulfuric acid; N = nitric acid; Z = zinc acetate; M = methanol; H = hydrochloric acid

1. TO MEET REGULATORY REQUIREMENTS, THIS FORM MUST BE COMPLETED IN DETAIL AND INCLUDED IN THE SHIPPER CONTAINING THE SAMPLES DESCRIBED.
2. PLEASE USE ONE LINE PER SAMPLE, NOT PER BOTTLE.
3. RETURN THIS FORM WITH SAMPLES • CLIENT MAY KEEP PINK COPY.
4. PARTIES COLLECTING SAMPLE LISTED AS REPORT TO AND LISTED AS ... AGREE TO STANDARD TERMS & CONDITIONS ON REVERSE.

VOCs, GRO/PVOC SAMPLING INSTRUCTIONS
for
WATER

Caution!

Bottles marked "HCl Preservative" contain small quantities of Hydrochloric acid. Avoid ingestion and skin contact. Flush exposed skin area with water immediately. Flush eyes 15 minutes and get medical attention. If ingested seek medical attention immediately. Appropriate MATERIAL SAFETY DATA SHEETS (MSDS) are included with each bottle shippment.

PROPER SAMPLE COLLECTION:

The client must fill at least two VOC vials with the water to be sampled. Each vial contains a small amount of Hydrochloric Acid which is used to preserve the sample. Do not discard this preservative.

1. Take at least two VOC vials and label them with sampling information including: date, time and the site location description.

2. Slowly fill the vials to a point <u>just before they overflow</u> the container. Avoid turbulence while filling.

3. When filled correctly a small dome of water will appear on the top of the vial.

4. Carefully place the cap on the vial and tighten it securely.

5. Invert the vial and tap on the cap to check for air bubbles.

NOTE: If bubbles are present, uncap the vial and add a little more water to again form the small dome of water. Carefully cap and seal the vial again. The EPA and Wisconsin DNR indicate that very small bubbles do not significantly impact VOC results.

6. Fill the other VOC vial(s) using steps 2 through 5.

7. Place vials on ice to cool them to 4 degrees C. Ship the samples back to Northern Lake Service via United Parcel Service - ground transportation. U.P.S. provides Northern Lake Service with one day service throughout Wisconsin, the U.P. of Michigan, and most of Minnesota. Samples shipped Monday through Thursday will arrive at NLS the next morning.

Please call Northern Lake Service if you have any questions. (715) 478-2777.

/vocsamp.ins

NLS SAMPLE TRACK

QUICK TURN	DUE DATE

CLIENT:	ORDERED BY:	DATE:
	PHONE:	ORDER REC'D BY:
	DESCRIPTION/CLIENT PROJ:	DATE TO BE SAMPLED:
☐ SHIPPING ADDRESS ON REVERSE SIDE		
DATA TO:	EXTRA COPIES:	BILLING TO:
TAD TO:	DONE ☐	
PRICING:		QUOTE #:
		PO #:
		MOB:
		PURG: TAD/DISK:

C.O.C. #	DATE COLLECTED	DATE REC. AT NLS	SAMPLE MATRIX	NLS SAMPLE #S	NLS PROJ. #s	BOTTLES RCV'D

LAB INSTRUCTIONS:	REPORTING INSTRUCTIONS:	SAMPLE CONDIT / COMMENTS:
	TAD REQUIRED (type) _____ ☐	
	SPECIAL REQUIREMENTS: _____	

CLIENT CONTACT AFTER SAMPLE RECEIPT:

WHO	FOR	INIT	DATE
	Delivery		
	Log-in		
	Log-in Check		
	Log-in Check		
	Subbed Out		
	Work Review		
	Work Review		
	Special Report		
	TAD / Disk		
	Data / Package Review - 1		
	Data / Package Review - 2		
	Final Pkg. Check & Copied		
	Billing Check		

NORTHERN LAKE SERVICE, INC.

EXAMPLE SAMPLE TAGS (LABELS)

Sample Label for Volatile Organic Compounds – Method 8260

NORTHERN LAKE SERVICE, INC.
400 N. Lake Ave., Crandon, WI 54520
715-478-2777 WI Lab Cert. #721026460

CLIENT _____
SAMPLE ID _____
DATE _____ TIME _____
☐Grab ☐Composite ☐Filtered ☐Unfiltered

CAUTION!

HCl
Acid

Sample Label for Semi-Volatile Organic Compounds – Method 8270

NORTHERN LAKE SERVICE, INC.
400 N. Lake Ave., Crandon, WI 54520
715-478-2777 WI Lab Cert. #721026460

NO PRESERVATIVES ADDED

CLIENT _____
SAMPLE ID _____
DATE _____ TIME _____
☐Grab ☐Composite ☐Filtered ☐Unfiltered

Sample Label for Cyanide – Method 335.4

NORTHERN LAKE SERVICE, INC.
400 N. Lake Ave., Crandon, WI 54520
715-478-2777 WI Lab Cert. #721026460

CLIENT _____
SAMPLE ID _____
DATE _____ TIME _____
☐Grab ☐Composite ☐Filtered ☐Unfiltered

CAUTION!

NaOH

NORTHERN LAKE SERVICE, INC.

EXAMPLE SAMPLE TAGS (LABELS)

Sample Label for Metals in Water– Nitric Acid

Sample Label for Mercury in Water – Hydrochloric Acid

Sample Label for Hexavalent Chromium in Water – Non Preserved